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ANALYSES OF EXPLOSIVES. PHYSICAL-CHEMICAL DATA, CHROMATOGRAMS, --ETC(U)
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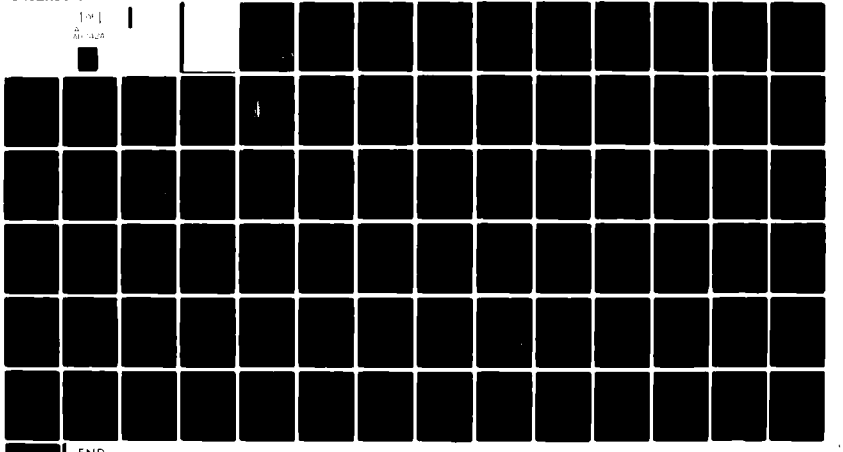

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NATIONAL DEFENCE RESEARCH INSTITUTE
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FOA-Report
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ANALYSES OF EXPLOSIVES

PHYSICAL-CHEMICAL DATA, CHROMATOGRAMS, MASS-, IR- AND NMR-SPECTRA. COLOUR REACTIONS AND THIN LAYER CHROMATOGRAPHY

Anita Alm, Olof Dalman, Inger Frölén-Lindgren,
Felix Hultén, Tom Karlsson and Monica Kowalska

81 pp

Summary

The present report comprises analytical data of frequently used explosives and tri-, di- and mononitrotoluenes. The data given are obtained by instrumental methods such as MS (70 eV, 20 eV, CI), IR and NMR (^{1}H, ^{13}C), in the form of spectra, and HPLC and GC, as chromatograms. Some data of noninstrumental methods such as colour reactions, thin layer chromatographic applications and pertinent physical-chemical data of these substances are also given.

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INTRODUCTION

Literature searches[1,2] on identification and analyses of explosives[3] have shown that there is no comprehensive report on analytical methods for this group of substances.(A recently published review is Yinon, J: Analysis of explosives. CRC Crit Rev Anal Chem 7 (1977) 1-35.)

Due to this fact the present work was carried out. The report comprises spectra and chromatograms of 25 substances of which eleven are frequently used explosives and the remaining fourteen are nitrotoluene isomers.

Instrumental methods have been applied. The spectral analyses include mass spectrometry (electron impact, chemical ionization), infrared spectrometry and nuclear magnetic resonance (^{1}H, ^{13}C). The chromatographic analyses include gas chromatography and high pressure liquid chromatography. Data on instrumentation and experimental conditions are given in each chapter.

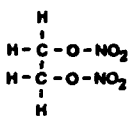
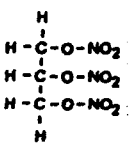
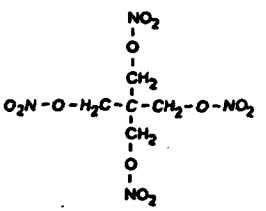
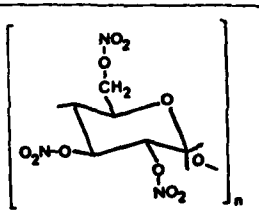
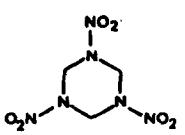
As a complement, some pertinent physical-chemical data are presented in tabulated form. Data on qualitative analyses are given, based on noninstrumental methods such as colour reactions and thin layer chromatography.

The collection of data presented will hopefully be of assistance in the qualitative and quantitative analysis of the components in explosive compositions.

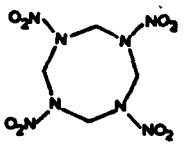
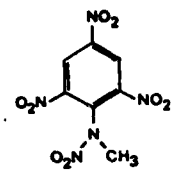
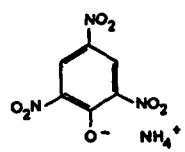
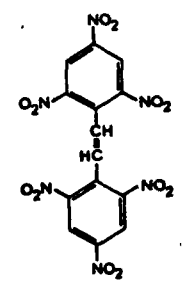
PHYSICAL DATA

Physical data given in tables 1-3 have been collected from different sources: compilations such as Landholt-Börnstein, Beilstein, Handbook of chemistry and physics, Fedoroff[4], books such as Meyer[5], Bofors[6] and a few published papers.

Table 1. List of explosives. Formulas and physical data.

No	Explosive	Abbrev	Synonym	Formula	Mol.-weight	Melt.-point	Deflagr. temp. °C	Remark
1	Ethylene glycol dinitrate	EGDN	(Di)nitroglykol, Glykoldinitrat		152,1	-22	217	
2	Nitroglycerine	NG	Glycerin- el glyceroltrinitrat, Trinitroglycerin		227,1	13,5 2,8	223-25	Modifications: 13,5 °C triclinic, stable 2,8 °C dipyramidal, rhombic, labile
3	Pentaerythritol tetranitrate	PETN	Pentaerytritel Pentaerytritol-tetranitrat, Pentrit, Pentryl, Penta, Nitropenta (-erytrit)		316,2	141,3	202-05	
4	Nitrocellulose	NC	Bomullskrut Cellulosanitrat		~ 10^5	-	185-190	
5	Hexogen	RDX	Cyclonit, T4, 1,3,5-Trinitros-triazin, 1,3,5-trinitro-1,3,5-triazacyklohexan		222,1	202	230	Decomposition close above the melting point

contin. Table 1

No	Explosive	Abbrev.	Synonym	Formula	Mol.-weight	Melt.-point	Deflagr. temp. °C	Remark
6	Octogen	HMX	Homocyclonit, 1,3,5,7-Tetranitro-1,3,5,7-tetraazacyklooktan		296,2	282	290	Modifications: (α orthoromb) β monocline (γ monocline) (δ hexagonal)
7	Tetryl	TETR	CE, 2,4,6,N-Tetranitro-N-metylanilin		287,2	131,5	185-195	Melts with decomposition
8	Ammonium picrate	AM-PIKR	Explosive D, Ammonium-2,4,6-trinitrofenolat		246,1	265-71	320	Melts with decomposition
9	Hexanitrostilbene	HNS	2,2',4,4',6,6'-Hexanitrostilben		450	316		Decomposition at 280 °C: 5h 7 % 7h 26 % 8h 48 %

contin. Table 1

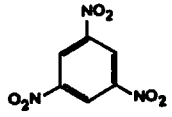
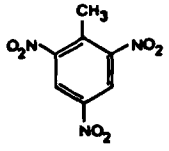
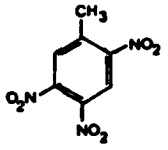
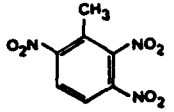
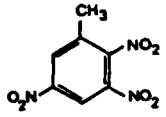
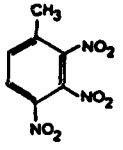
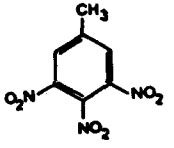
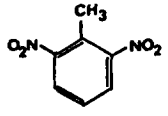
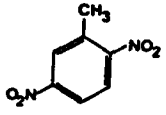
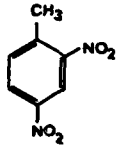
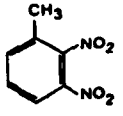
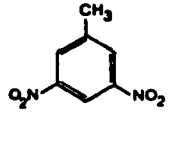
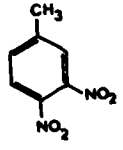
No	Explosive	Abbrev	Synonym	Formula	Mol.-weight	Melt.-point	Deflagr. temp. °C	Remark
10	Trinitroben-zene	TNB 1,3,5-TNB	Benzit, 1,3,5-Trinitro-bensen		213,1	123,2		
11	Trotyl, 2,4,6-Trinit-rotoluene	TNT 2,4,6-TNT	2,4,6-Trinitro-toluen Tri, Trotyl, Trolit, sym-TNT, α-TNT		227,1	80,8	300	Decomposition begins at 180 °C

Table 2. List of nitrotoluenes*. Formulas and physical data.

No	Compound	Abbrev	Synonym	Formula	Mol.-weight	Melt.-point
12	2,4,5-Trinitrotoluene	2,4,5-TNT	γ-TNT	 12	227,1	104
13	2,3,6- "	2,3,6-TNT	η-TNT	 13		111
14	2,3,5- "	2,3,5-TNT	ε-TNT	 14		97,2
15	2,3,4- "	2,3,4-TNT	β-TNT			112
16	3,4,5- "	3,4,5-TNT	δ-TNT	 15  16		137,5
17	2,6-Dinitrotoluene	2,6-DNT			182,1	66
18	2,5- "	2,5-DNT		 17		52,5
19	2,4- "	2,4-DNT		 18		70,1
20	2,3- "	2,3-DNT		 19		61
21	3,5- "	3,5-DNT				93
22	3,4- "	3,4-DNT		 20  21  22		59,8

*2,4,6-Trinitrotoluene, see no 11, table 1.

Contin. Table 2

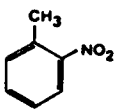
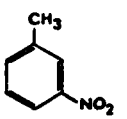
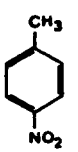
No	Compound	Abbrev	Synonym	Formula	Mol.-weight	Melt.-point
23	2-Mononitrotoluene	2-MNT	o-nitro-toluen	 23	137,1	{-10,6(α) {- 4,1(β)
24	3- "	3-MNT	m- "	 24		15,5
25	4- "	4-MNT	p- "	 25		51,3

Table 3. Solubilities of explosives (g/100 g at 25 °C unless specified)

Solvent / Explosive	Acetone	Aceto-nitrile	Benzene	Butyro-lactone	Dimethyl-formamide	Dimethyl-sulph-oxide	Ethanol	Ether	Ethyl-acetate	Chloro-form	Carbon-disul-phide	Carbon-tetra-chloride	Toluene	Water
1. EGDN	∞		∞				v s	∞		∞	sl s	ca 2 vol%	∞	0,56
2. NG	∞		∞				54 (20°C)	∞	∞	∞	1,25 vol%	2 vol%	v s	0,15 (20°C)
3. PETN	20,3 (20°C)		0,3 (20°C)				0,20 (20°C)	0,25 (20°C)	6,3 (19°C)	0,06 (19°C)			0,23 (20°C)	sl s
4. NC[1]	v s													
5. RDX	8,2	5,5	0,05 (20°C)	14	37	41	0,11 (20°C)	0,055 (20°C)	1,5 (20°C)	0,008 (20°C)	sl s	0,005 (50°C)	0,02 (20°C)	0,006
6. HMX	2,8	2,0		12	2,3[2] (20°C)	57								sl s
7. TETR	68 vol%		3,5 (20°C)		114		0,65	0,46	12,2 (18°C)	0,68	0,024	0,031	3,0 (19,5C)	0,008
8. AM-PIKR					90	75	0,62 (24°C)	0,004 (24°C)						1,1 (20°C)
9. HNS	< 0,1			0,4	1,5	1,4	s							sl s
10. TNB(17°C)	59		6,2				2,1	1,7	30	6,2	0,24	0,24	12	0,03 (15°C)
11. TNT	132		88		142	128	1,5	3,8		25	0,63	0,82	67	0,02

1) Ketones, esters and ether + ethanol are common solvents for nitrated cellulose. The solubility depends on degree of nitration.

2) Solvate crystals precipitate.

CHROMATOGRAPHY

High pressure liquid chromatography (HPLC)

High pressure liquid chromatography is used in qualitative as well as in quantitative analysis[2]. Substances of low vapour pressure or of thermal instability are often more easy to analyse by means of high pressure liquid chromatography than by gas chromatography.

The instrumentation used was a Waters Assoc liquid chromatograph, model ALC-GPC-204, equipped with a 254 UV detector, model 440. A μ-Porasil column was used (1/4" x 1", 10 μ, 400 m^2/g, Waters Assoc). Eluents were of Uvasol (Merck's) purity. The sensitivity of the detector is approximately of the same order for the aromatic nitro compounds and the nitramines but some ten powers lower in the case of the nitric esters[7].

Retention volumes of the various explosives are given in table 4. A solvent peak emanating from the sample injected may appear in a HPLC chromatogram. In case the peak is of considerable height it has been indicated.

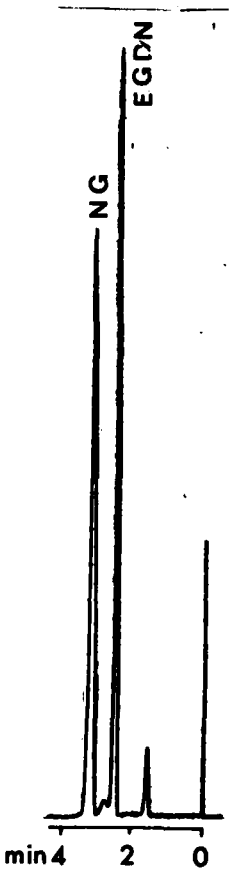
Reversed phase chromatography (Lichrosorb C2 column and water-ethanol as eluent) resulted in chromatograms with less resolved peaks.

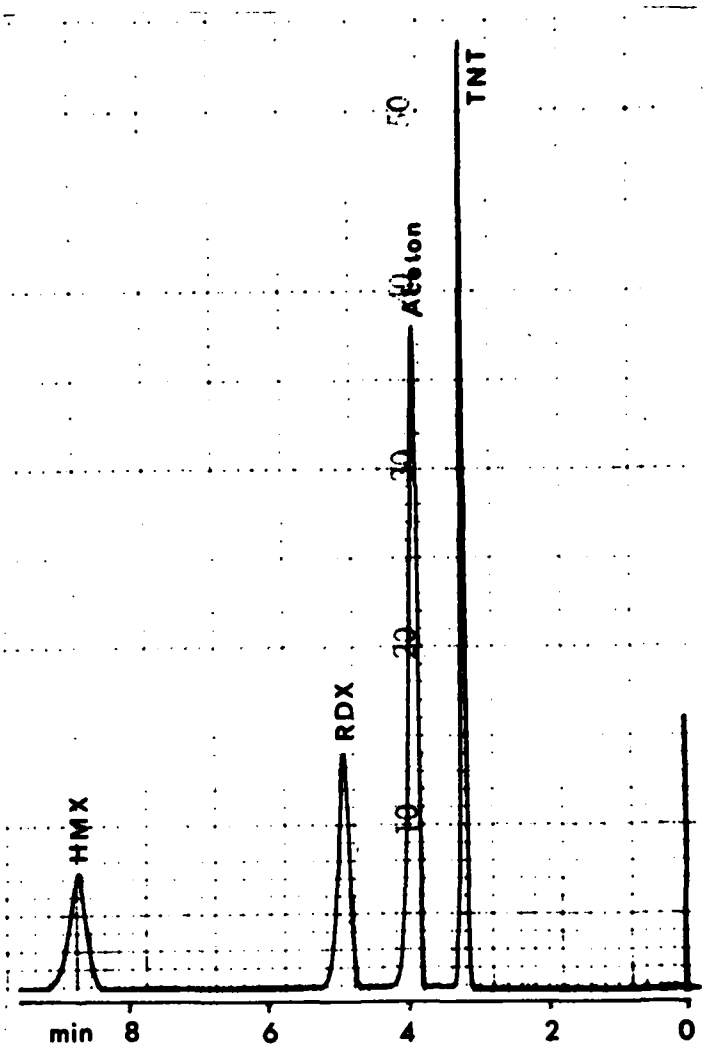
Table 4. Retention volumes (ml) of explosives on a μ-Porasil column. V_M = 3,0 ml (t = tailing).

Substance \ Eluent	1	2	3	4	5
11. TNT	3,1	3,1	3,3	5,6	5,0
1. EGDN	3,2	3,3	3,8	4,8	6,1
2. NG	3,2	3,4	4,0	5,2	7,0
10. TNB	3,1	3,3	4,0	7,3	7,5
3. PETN	3,2	3,2	7,0	15,6	8,6
7. TETR	3,3	3,6	7,0	15,9	31,0
9. HNS	3,2	3,2	3,6	> 40	> 40
5. RDX	4,8	11,4	> 40	> 40	
6. HMX	8,6	> 40	> 40		
$CHCl_3$			3,0	3,3	3,2
Acetone	3,7	3,8	3,9	t	t

Eluents:

1. Chloroform-acetonitrile 90:10
2. Chloroform
3. c-Hexane-chloroform 50:50
4. c-Hexane-dichloromethane 70:30
5. c-Hexane-chloroform 80:20





Column:
μ-Porasil

Eluent:
c-Hexane-dichloromethane, 70:30

Flow rate:
2,0 ml/min

Sample volume:
10 μl

Sample conc:
Totally 2 % in ethanol
EGDN:NG 1:1

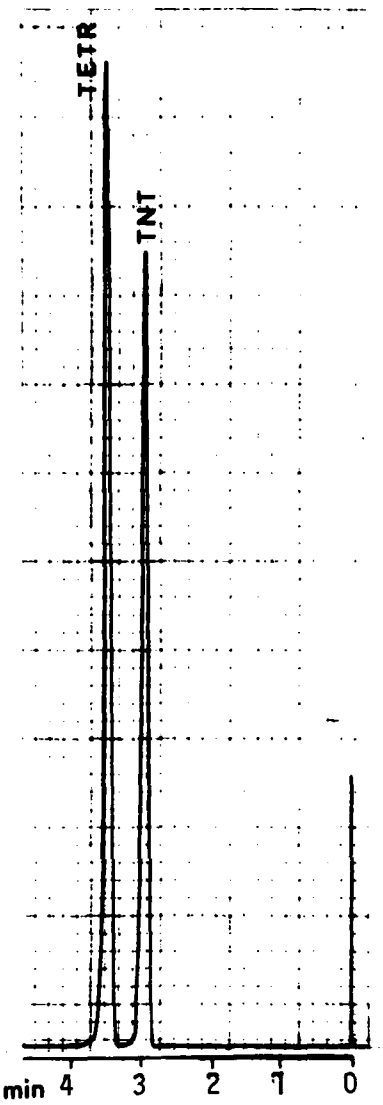
Column:
μ-Porasil

Eluent:
Chloroform-acetonitrile, 90:10

Flow rate:
1,0 ml/min

Sample volume:
3 μl

Sample conc:
Totally 0,6 % in acetone
TNT:RDX:HMX 1:1:1



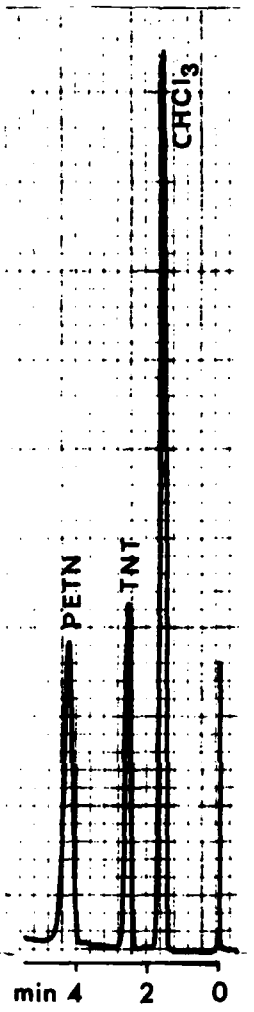
Column:
μ-Porasil

Eluent:
Chloroform

Flow rate:
1,0 ml/min

Sample volume:
10 μl

Sample conc:
Totally 0,07 % in chloroform
TNT:TETR 3:4



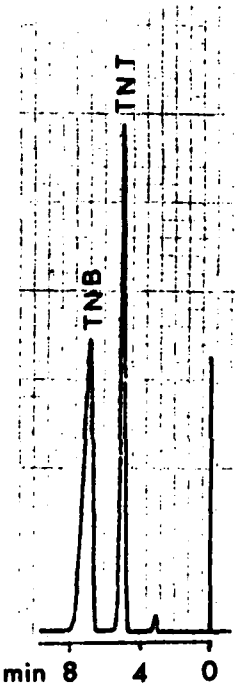
Column:
μ-Porasil

Eluent:
c-Hexane-chloroform,
80:20

Flow rate:
2,0 ml/min

Sample volume:
20 μl

Sample conc:
Totally 0,03 % in
chloroform
TNT:PETN 1:300



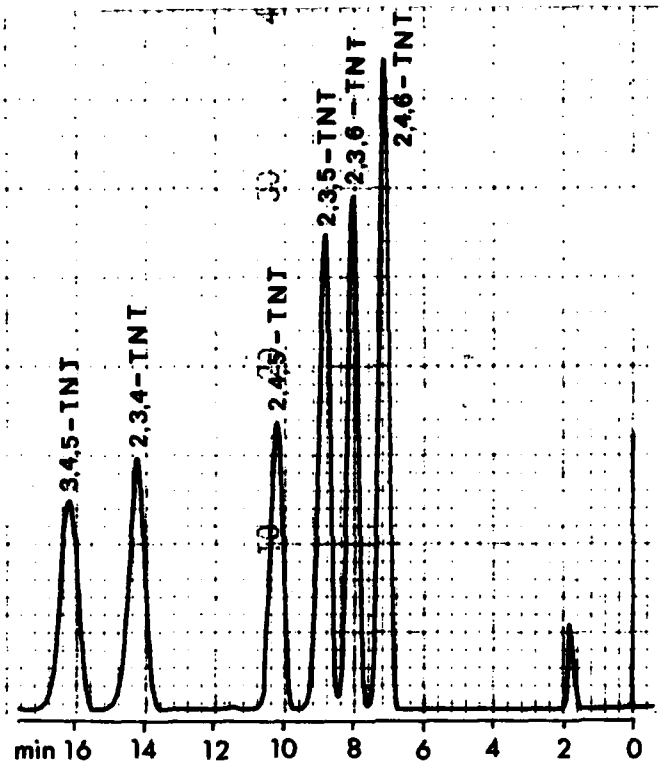
Column:
μ-Porasil

Eluent:
c-Hexan-chloroform,
80:20

Flow rate:
1,0 ml/min

Sample volume:
20 μl

Sample conc:
Totally 0,01 % in
chloroform
TNT:TNB 1:1



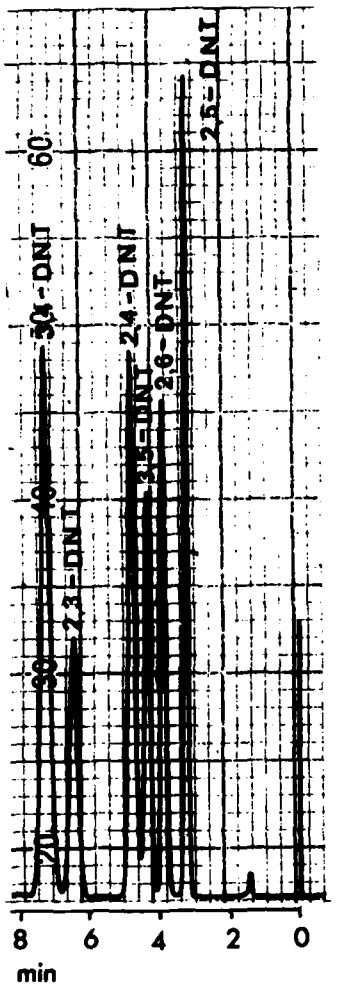
Column:
µ-Porasil

Eluent:
c-Hexan-dichloromethane, 87,5:12,5

Flow rate:
2,0 ml/min

Sample volume:
20 µl

Sample conc:
Totally 0,02 %



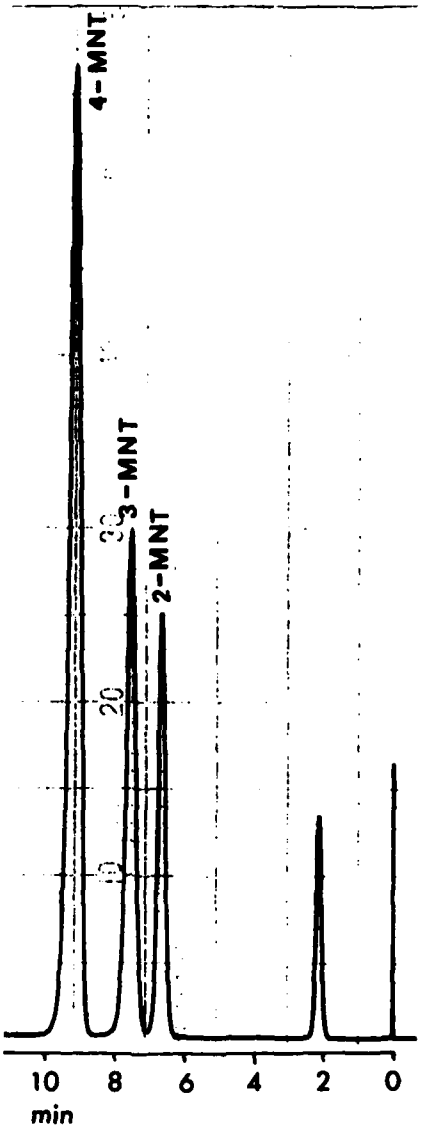
Column:
µ-Porasil

Eluent:
c-Hexan-chloroform, 95:5

Flow rate:
2,5 ml/min

Sample volume:
10 µl

Sample conc:
Totally 0,1 %



Column:
µ-Porasil

Eluent:
c-Hexane

Flow rate:
1,0 ml/min

Sample volume:
10 µl

Sample conc:
Totally 0,05 %

Gas chromatography (GC)

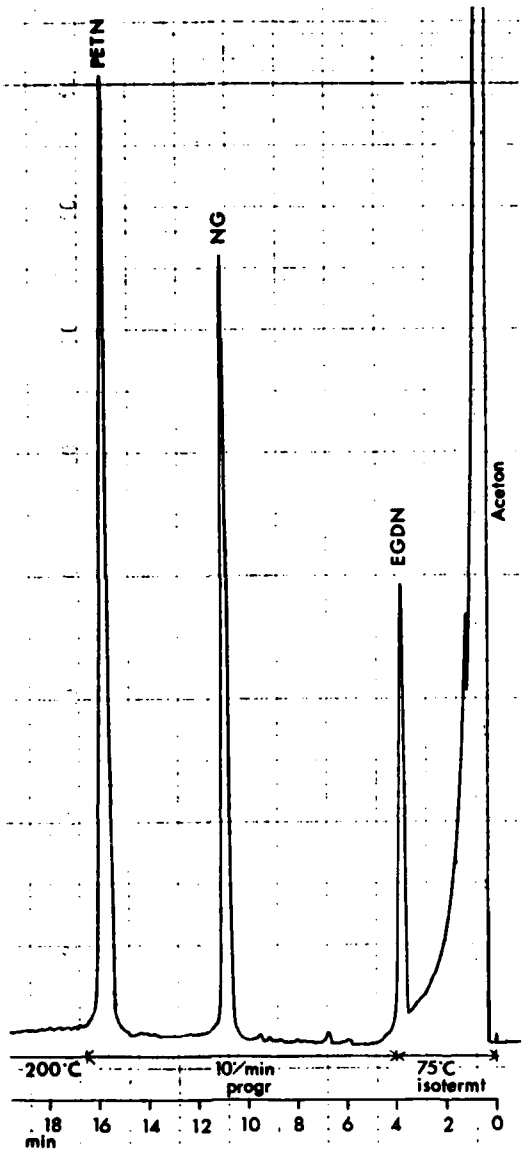
Gas chromatography is used in qualitative as well as in quantitative analysis and has been applied in analyses of hexotol samples[8].

The instrument used was a Varian gas chromatograph model 3700, equipped with a flame ionization detector. Columns of Pyrex glass were used, having OD 1/4", ID 1.5 mm, and the lengths of 0.75 m and 3 m. Data on column length, stationary phase, flow rates of gases, temperatures and sample size are given below each chromatogram. In all cases a sample volume of 1 µl was injected.

Because of their low vapour pressures the substances Nos 4(NC), 6(HMX), 8(AM-PIKR) and 9(HNS) were not chromatographed.

1 EGDN 3 PETN

2 NG



Column: 5% DC 550 on Chromosorb W-HP, 0,75 m x 1,5 mm

Temp:
Column: Progr Start: 75°C during 4 min, 10°/min
Final temp 200 °C

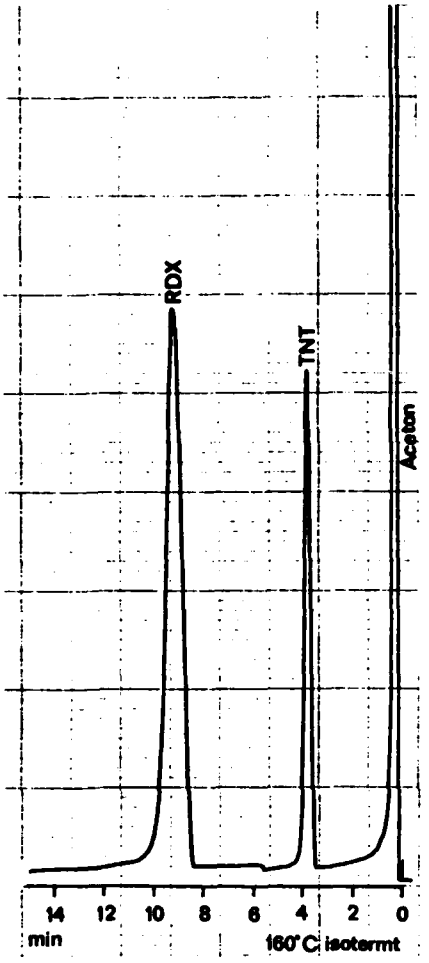
Inj: 130°C
Det: 220°C

Flow rate:
N_2 20 ml/min
H_2 20 ml/min
Air 250 ml/min

Sample conc: Totally 2 % in acetone
EGDN:NG:PETN = 1:2:3

18 11 TNT

5 RDX



Column: 5 % DC 550 on
Chromosorb W-HP
0,75 m x 1,5 mm

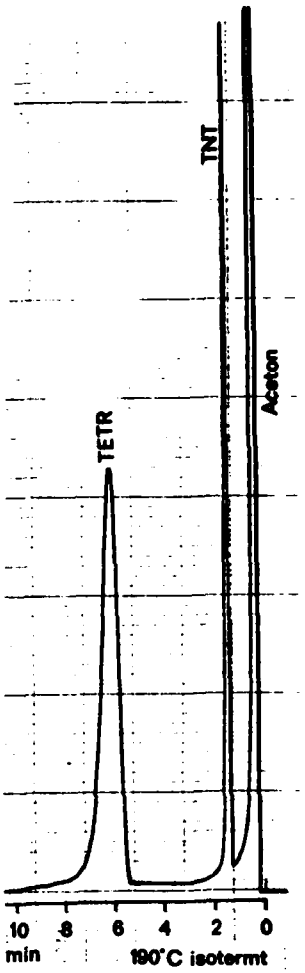
Temp:
Column: 160°C isotherm
Inj: 170°C
Det: 190°C

Flow rate:
N_2 20 ml/min
H_2 20 ml/min
Air 250 ml/min

Sample conc:
Totally 0,35 % in acetone
TNT : RDX = 2 : 7

11 TNT

7 TETR



Column: 5 % DC 550 on
Chromosorb W-HP
0,75 m x 1,5 mm

Temp:
Column: 190°C isotherm
Inj: 190°C
Det: 200°C

Flow rate:
N_2 20 ml/min
H_2 20 ml/min
Air 250 ml/min

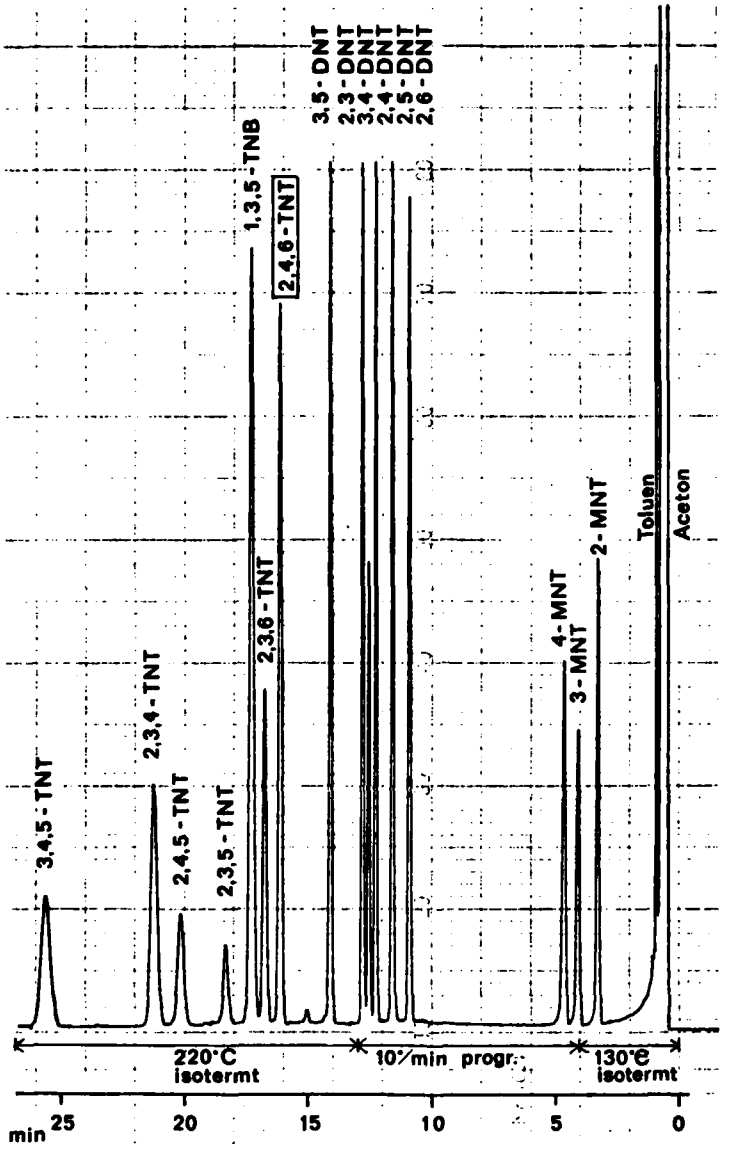
Sample conc:
Totally 0,5 % in acetone
TNT : TETRYL = 1 : 2

10 TNB

11 - 15 TNT

16 - 22 DNT

23 - 25 MNT



Column: 3 % OV 225 on Chromosorb W-HP, 3 m x 1,5 mm

Temp:
Column: Progr Start: 130°C during 4 min, 10°/min
Final temp 220 °C

Inj: 150°C
Det: 230°C

Flow rate:
N_2 20 ml/min
H_2 20 ml/min
Air 250 ml/min

Sample conc: Totally 0,5 % in acetone
MNT:DNT:TNT:TNB = 3:12:6:1

SPECTROSCOPY

The purity of the samples was verified by gas chromatography.

Mass spectrometry (MS)

The mass spectra were recorded with a LKB Model 2091 single focus spectrometer. The substances were analyzed by EI (electron impact)[9] at electron voltages of 70 and 20 eV and by CI (chemical ionization) with methane (1 Torr) as ionization media.

The majority of the samples were introduced into the mass spectrometer via a gas chromatograph. The columns used were identical to those specified in the chapter of gas chromatography. The substances Nos. 6(HMX) and 7(TETR) were introduced through the direct inlet, those of low vapour pressure Nos. 4(NC), 8(AM-PIKR), and 9(HNS) were not examined.

Data on mode of ionization, electron voltage, ion source temperature and characteristic fragments are given together with the spectrum.

Infrared spectroscopy (IR)

The IR spectra were recorded with a Perkin Elmer grating spectrometer model 377. Time constant: Auto; Scan mode: 13 min; Slit: N.

The majority of the samples were prepared in the form av a KBr disc composed of 0.5 - 1.8 mg substance in 300 mg KBr (diameter 13 mm, thickness 1 mm, applied pressure 1.500 kg/cm^2). The substances Nos. 1, 2, and 23 - 25 were dissolved in CCl_4 (0.1 - 0.2 M) and analyzed in a 0.12 mm NaCl-cell. Substance No. 4(NC) was analyzed as a film prepared from a solution of the sample in acetone.

Significant band assignments and sample concentration are given at each spectrum.

Table 5. IR-frequencies of (x)-NO_2 associated with nitric esters, nitramines and nitro compounds.

Substance group	(x)-NO_2(cm^{-1})	X
Nitric esters Nos. 1 - 4	1660 - 1640; 1285 - 1270	O
Nitramines Nos. 5 - 6	1590 - 1530; 1310 - 1270	N
Nitro compounds Nos. 7 - 25	1560 - 1520; 1370 - 1340	C

Nuclear magnetic resonance (NMR)

This method is used both in quantitative and qualitative analysis and has been applied in analyses of hexotol samples[13].

^{1}H NMR

Proton spectra[14] were obtained at 60 MHz on a Varian NV-14 spectrometer (CW). The internal reference was tetramethylsilane (TMS). The spectra were recorded with a sweep time of 500 seconds. Sample concentrations were 2 - 3 % in acetone-d_6*, except 9(HNS) and 4(NC) which were dissolved in dimethylsulfoxide-d_6** and 1(EGDN)dissolved in CCl_4. To 2(NG) acetone was added.

The chemical shifts of the different protons are indicated by letter symbols in formula and spectrum. Certain parts of the spectra have been expanded.

^{13}C NMR

Carbon-13 spectra[15] were obtained at 20 MHz with a Varian CFT 20 spectrometer system, equipped with a 10 mm probe. The pulse interval varied between 1 to 3 seconds. The internal reference was tetramethylsilane (TMS) and the spectra obtained are proton decoupled.

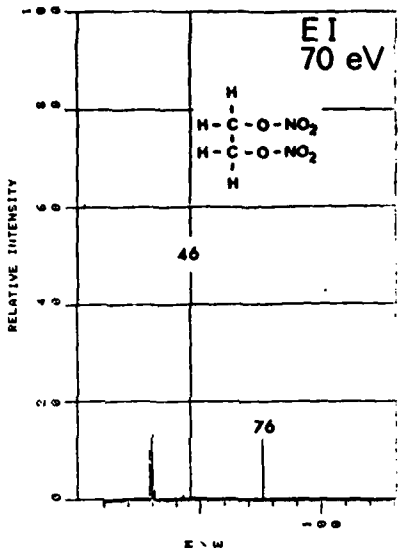
Sample concentrations were 3 - 8 % w/v in acetone-d_6, except 2(NG) and 9(HNS) which were 5 % in CCl_4 and 1 % in dimethylsulfoxide-d_6, respectively. To shorten the relaxation times, $Cr(acac)_3$ was added to a concentration of 3.6×10^{-2} M, except for 1 (EGDN) and 4(NC).

The line assignments are indicated by a letter at the corresponding carbon atom in the structure. The large solvent peaks from acetone-d_6 and dimethylsulfoxide-d_6 around 30 and 40 ppm, respectively, are not presented.

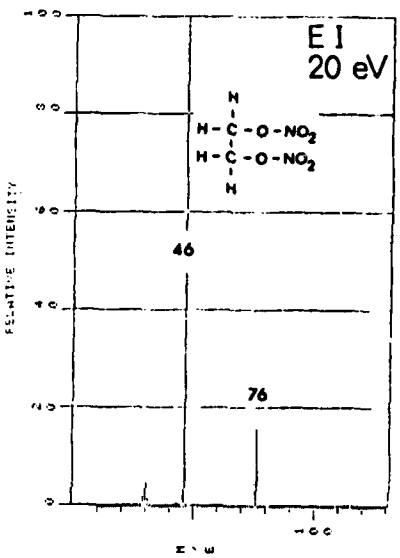
* Signal at 2.05 ppm

** Signal at 2.50 ppm

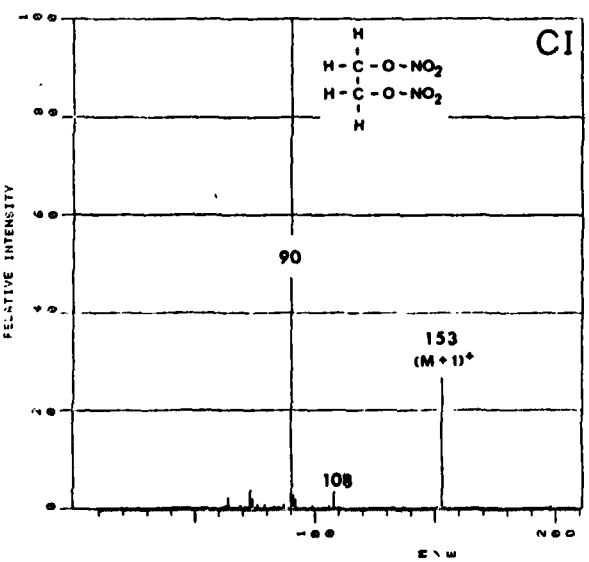
EGDN



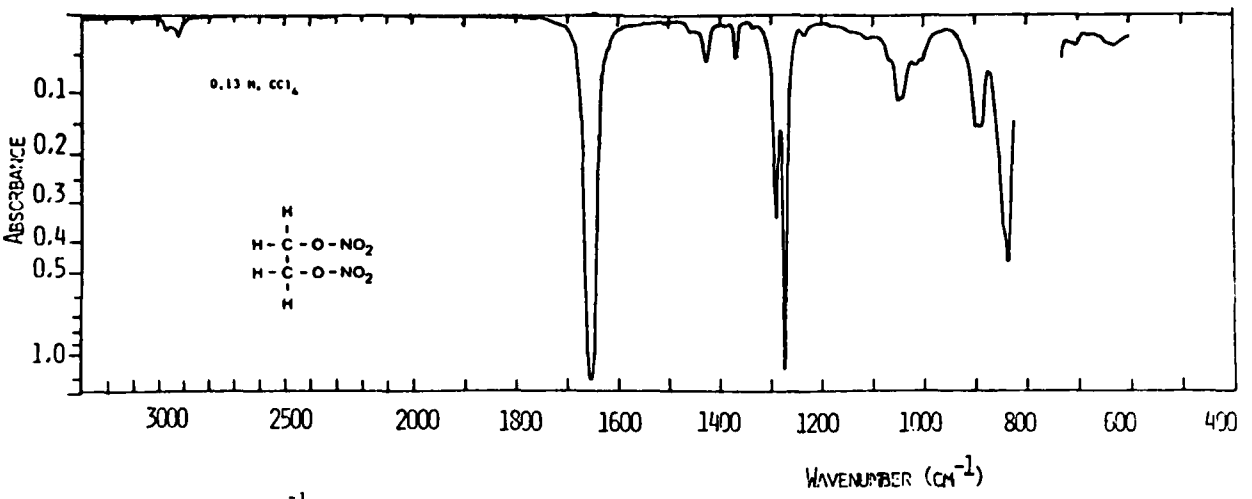
Inlet: GC
Column: DC 550
Ion source: 150 °C



Inlet: GC
Column: DC 550
Ion source: 150 °C



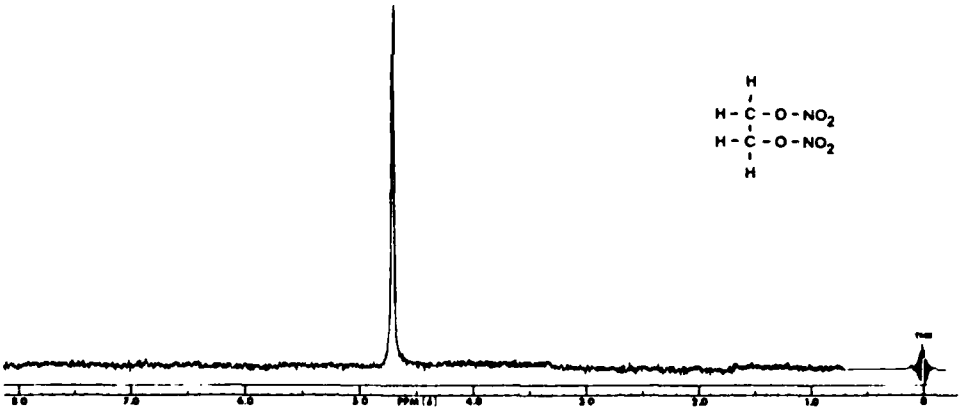
Inlet: GC
Column: DC 550
Ion source: 150 °C



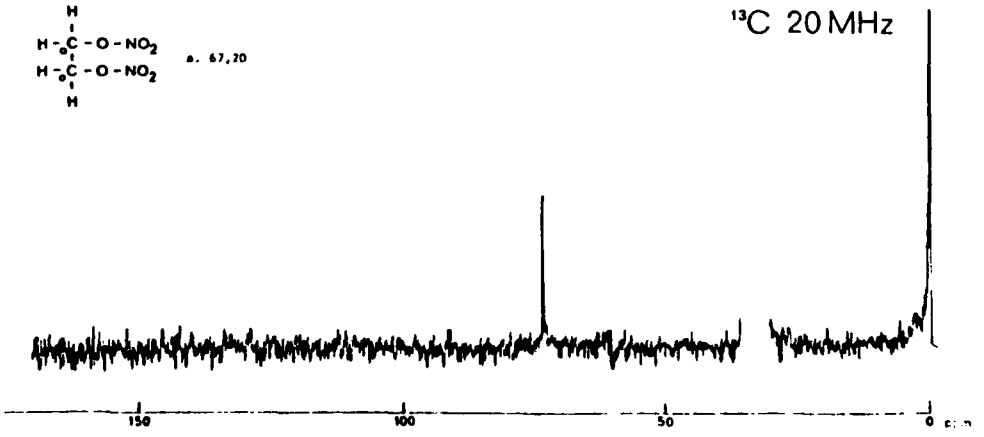
Group frequences (cm^{-1})

2965, 2930, 2920	C-H aliph	1460, 1428	C-H aliph	837	O-(NO_2)
1655	(O)-NO_2 asym	1270	(O)-NO_2 sym		

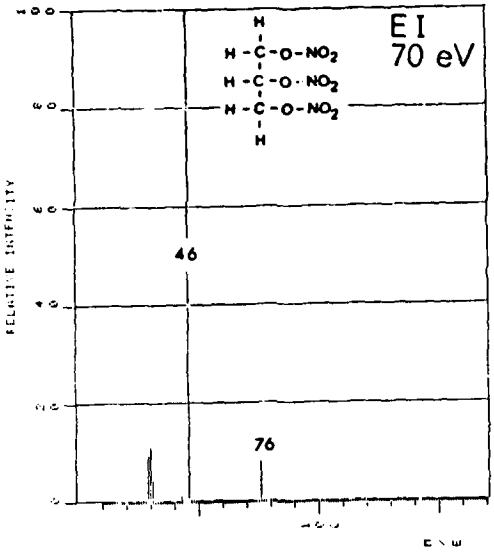
^1H 60 MHz



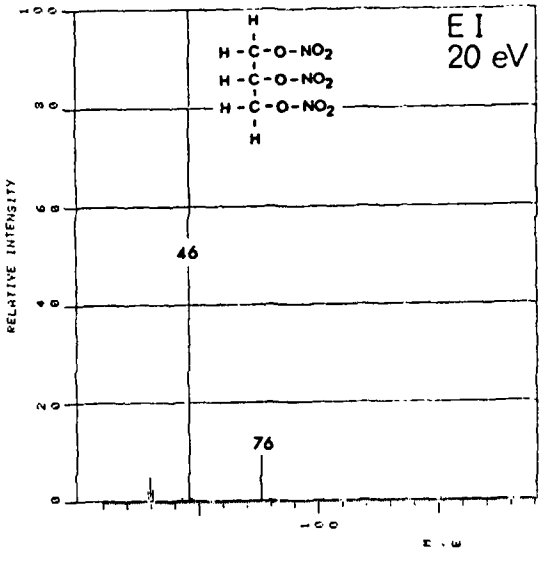
^{13}C 20 MHz



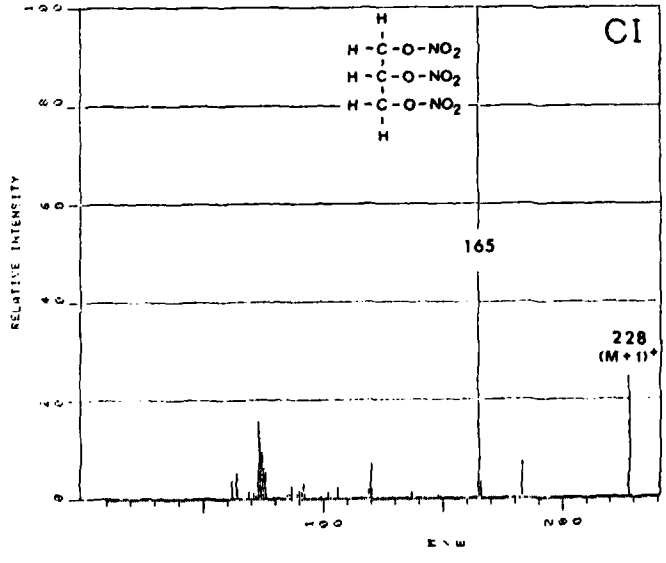
NG



Inlet: GC
Column: DC 550
Ion source: 150 °C

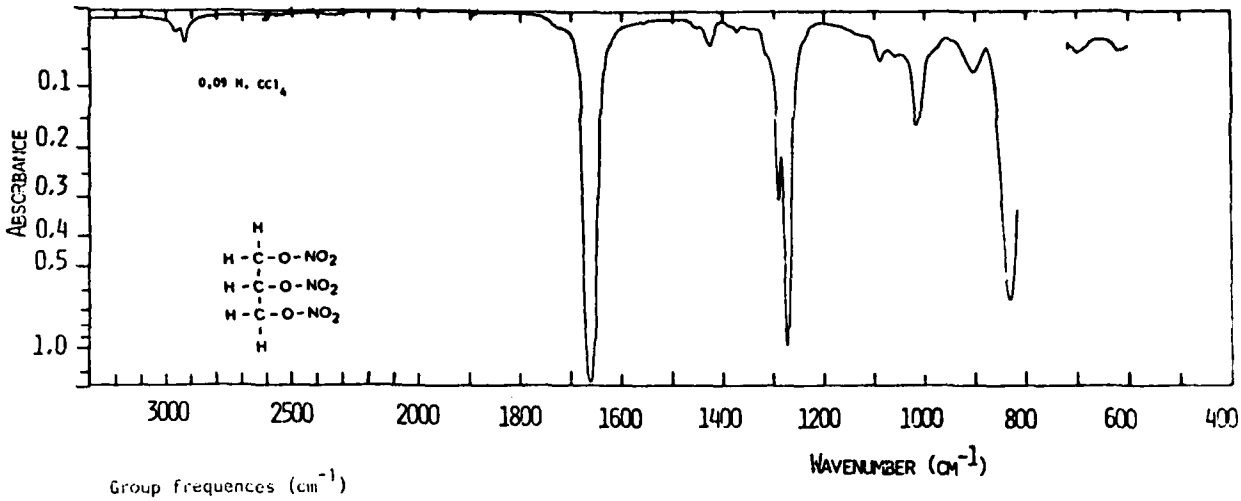


Inlet: GC
Column: DC 550
Ion source: 150 °C



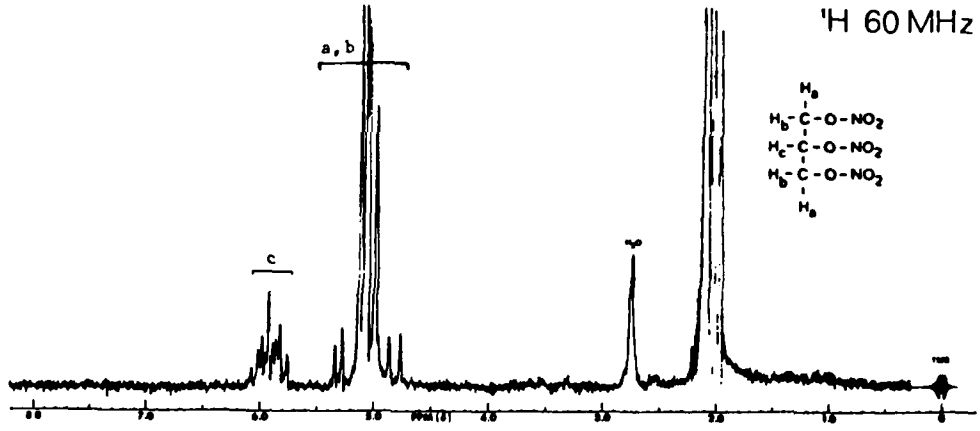
Inlet: GC
Column: DC 550
Ion source: 150 °C

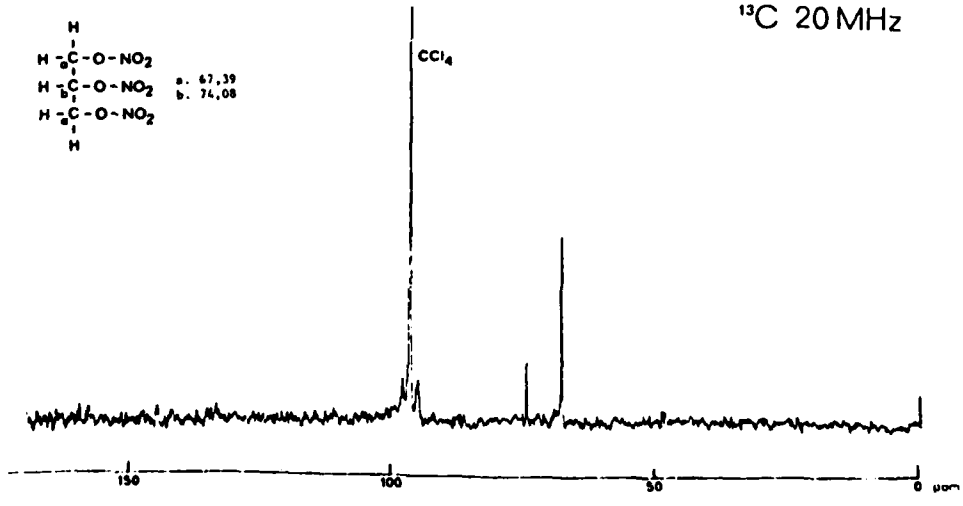
(2)



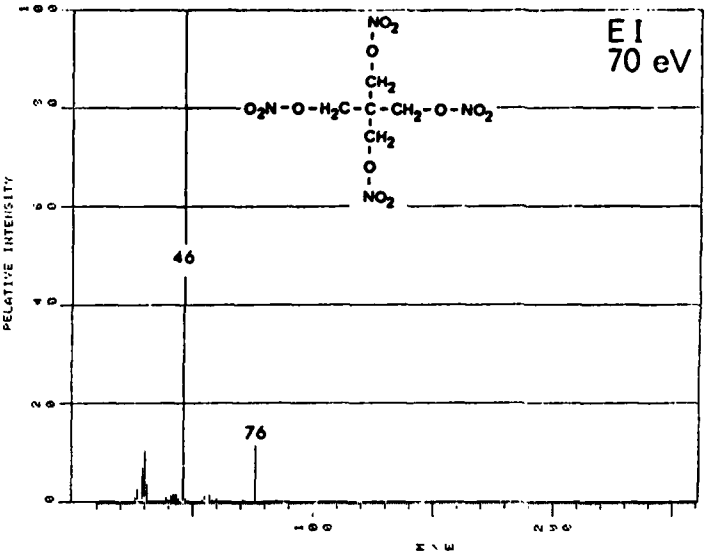
Group frequences (cm^{-1})

2961, 2923	C-H aliph	1424	C-H aliph	830	O-(NO_2)
1660	(O)-NO_2 asym	1272	(O)-NO_2 sym		

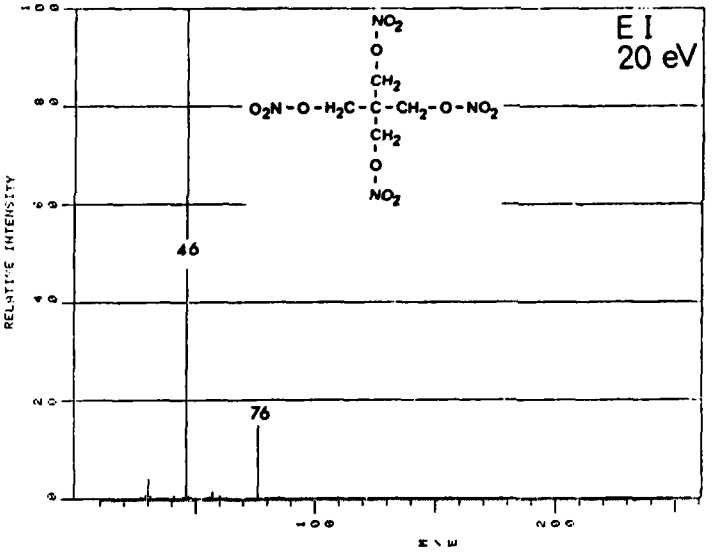




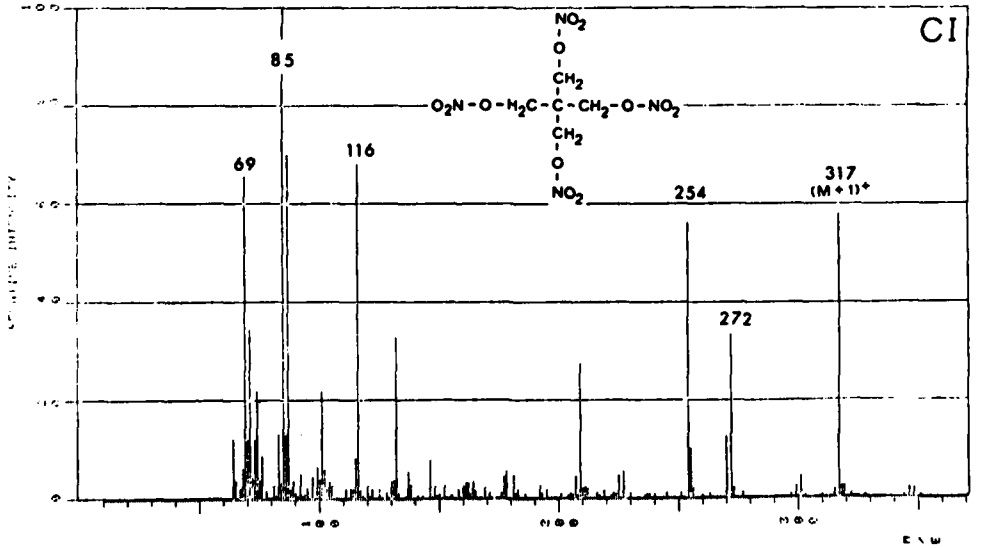
PETN



Inlet: GC
Column: DC 550
Ion source: 150 °C

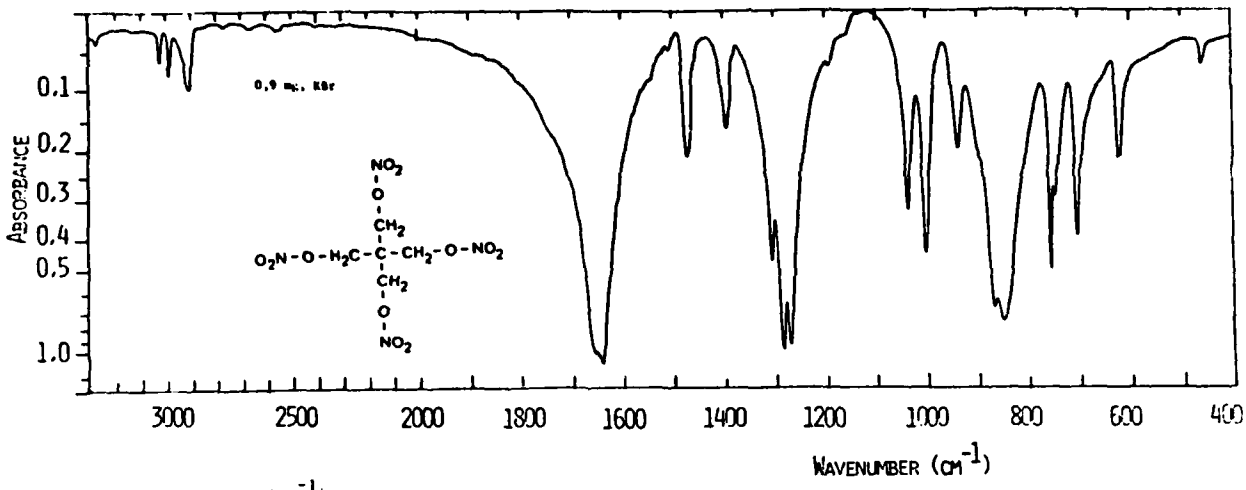


Inlet: GC
Column: DC 550
Ion source: 150 °C



Inlet: GC
Column: DC 550
Ion source: 150 °C

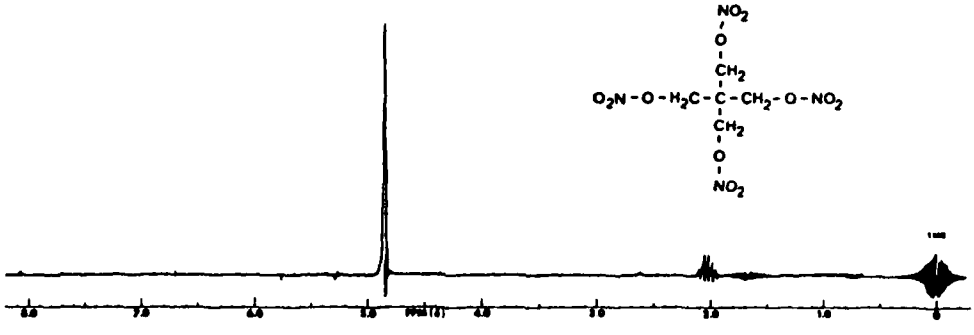
3

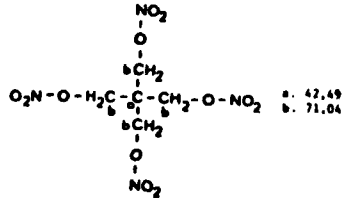


Group frequences (cm^{-1})

3280	Over tone from 1640	1660, 1640	(O)-NO_2 asym	1286, 1278	(O)-NO_2 sym	702	(O)-NO_2
3024, 2987	C-H aliph	1470, 1397	C-H aliph	870, 850	O-(NO_2)		

^1H 60 MHz





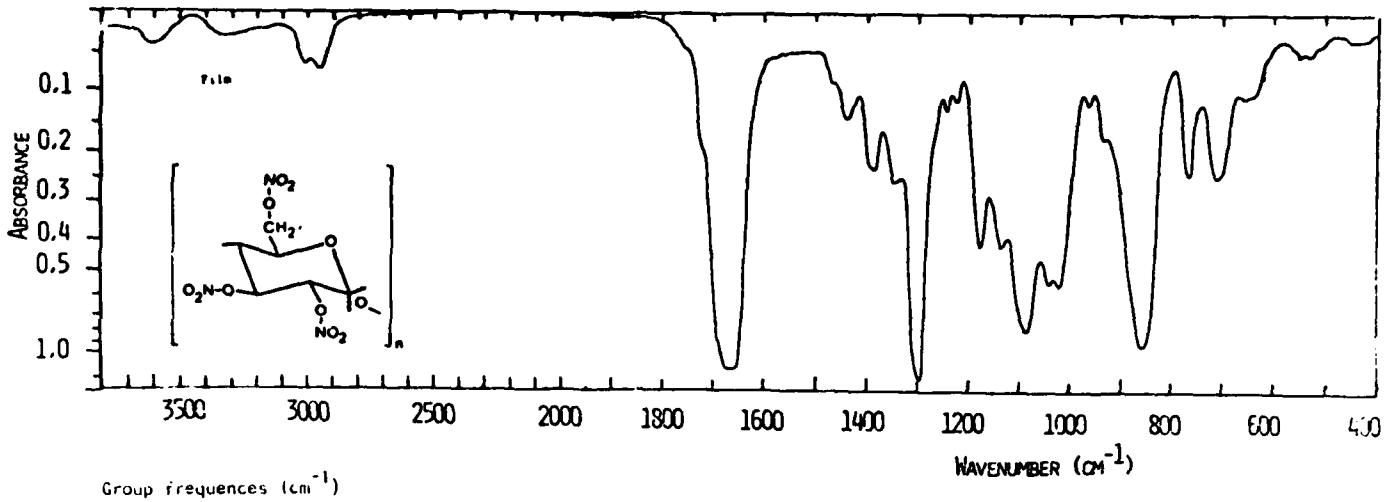
^{13}C 20 MHz

28

NC

See page 20.

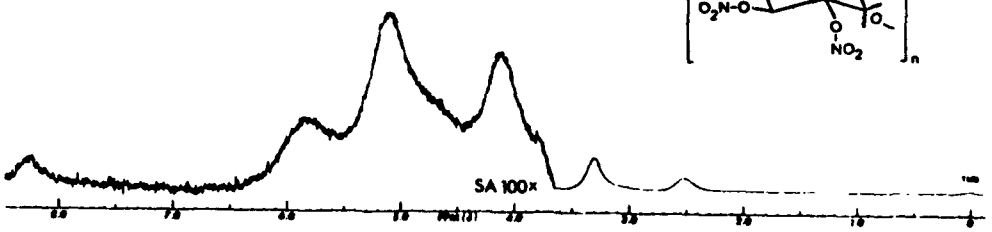
(4)



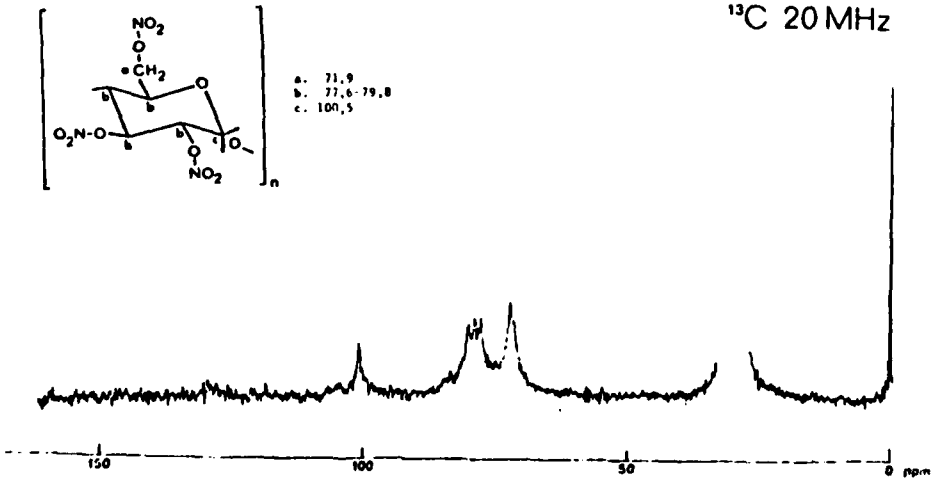
Group frequences (cm^{-1})

3600	OH free	3020, 2940	C-H aliph	1440	C-H aliph	1082	C-O-C	765, 705, 550	(O)-NO_2
3350	OH bound	1670, 1650	(O)-NO_2 asym	1295	(O)-NO_2 sym	855	O-(NO_2)		

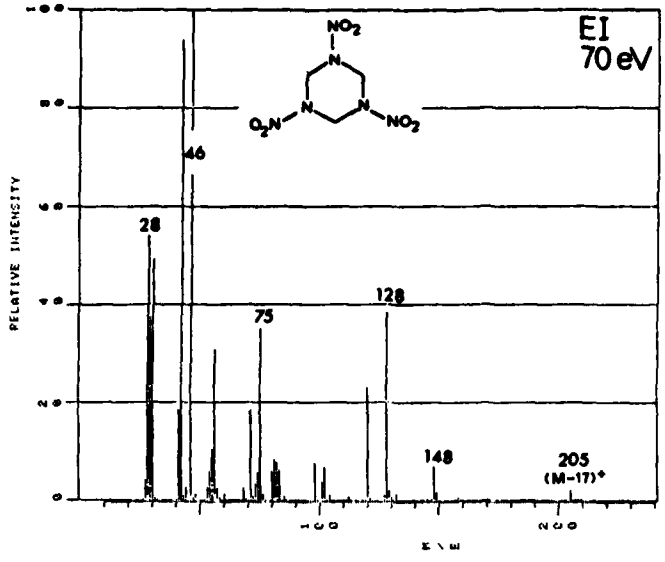
^{1}H 60 MHz



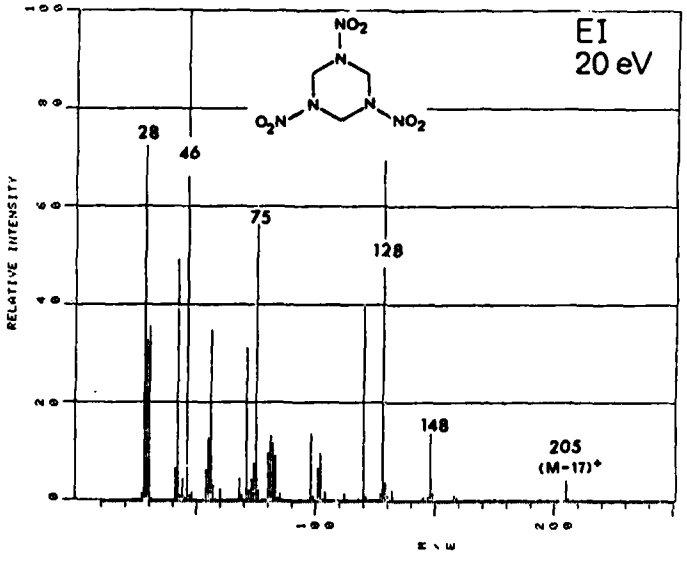
^{13}C 20 MHz



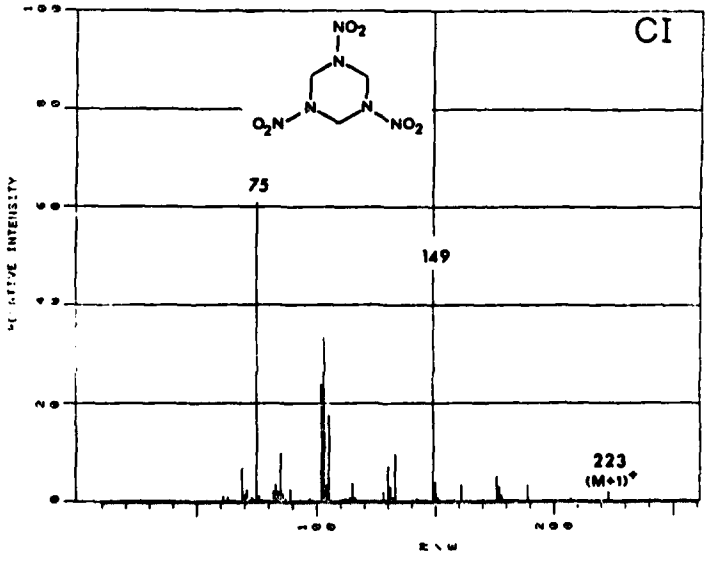
RDX



Inlet: GC
Column: DC 550
Ion source: 150 °C

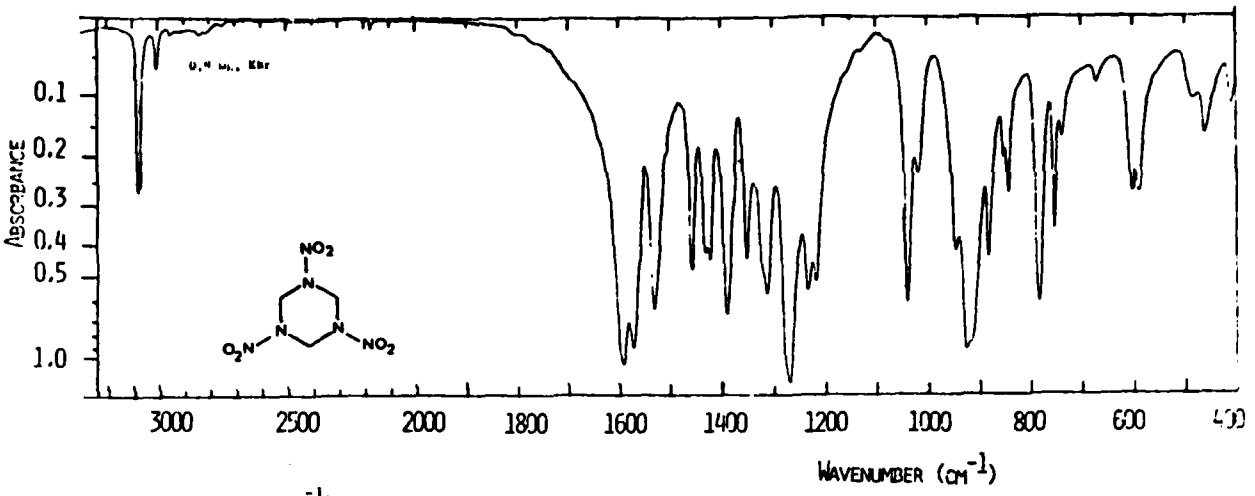


Inlet: GC
Column: DC 550
Ion soruce: 150 °C



Inlet: GC
Column: DC 550
Ion source: 150 °C

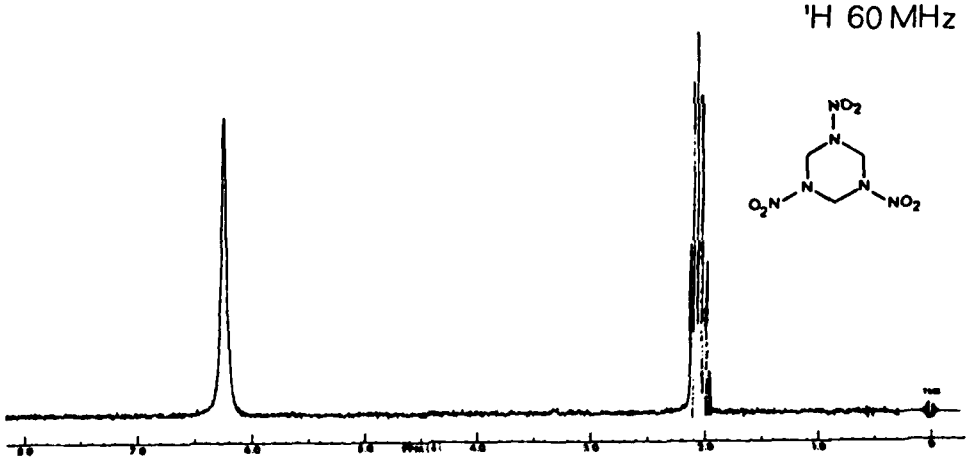
5



Group frequences (cm^{-1})

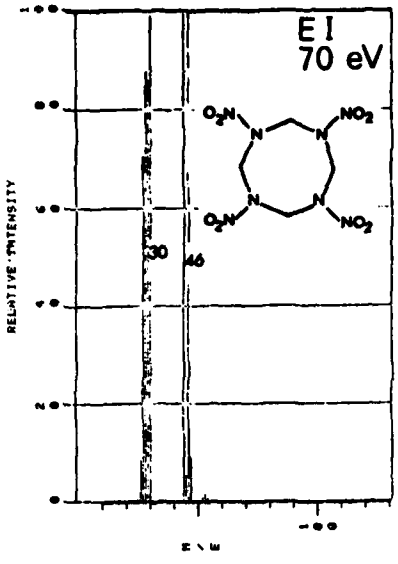
3079, 3069, 3002, 2951	C-H aliph	1459, 1389	C-H aliph	1040, 923	C-H
1590, 1570, 1531	(N)-NO_2 asym	1310, 1269	(N)-NO_2 sym	702, 602	(N)-NO_2

^1H 60 MHz

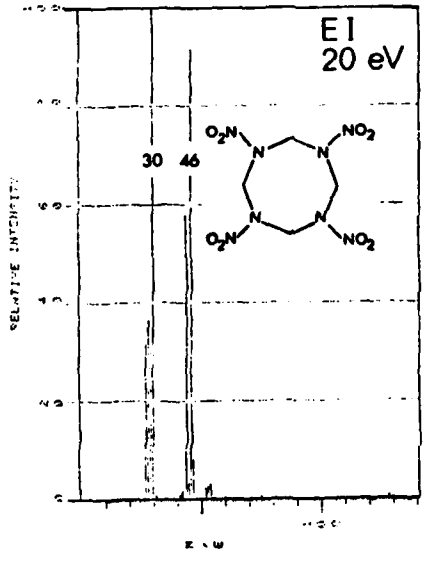


^{13}C 20 MHz

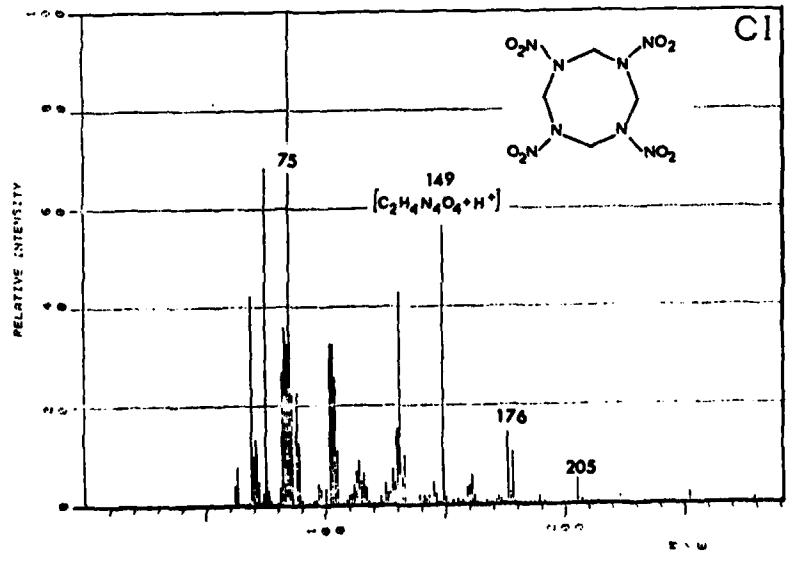
HMX



Inlet: Directly
Temp: 105 °C
Ion source: 150 °C

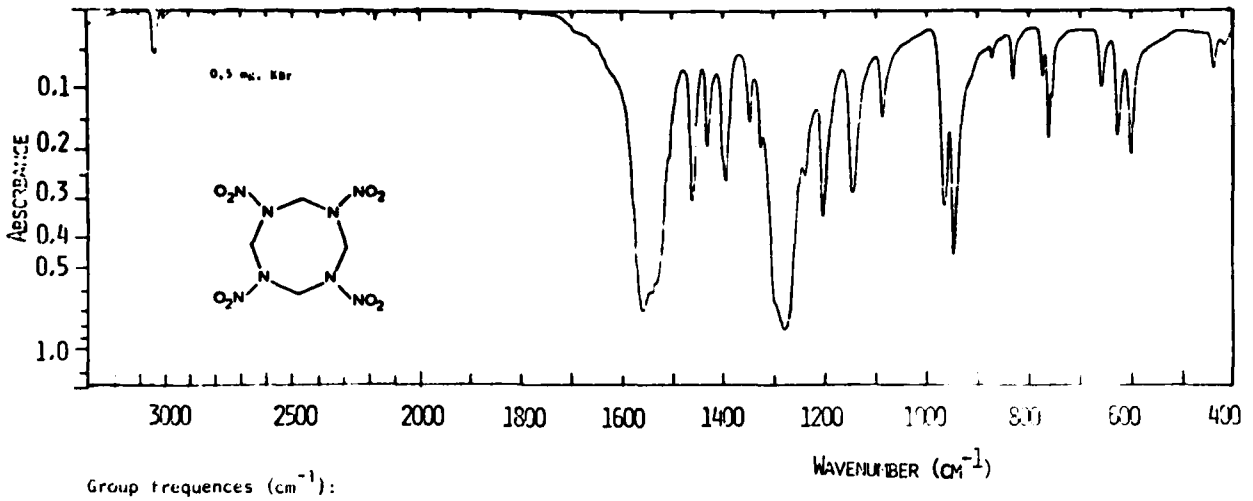


Inlet: Directly
Temp: 105 °C
Ion source: 150 °C



Inlet: Directly
Temp: 105 °C
Ion source: 120 °C

6

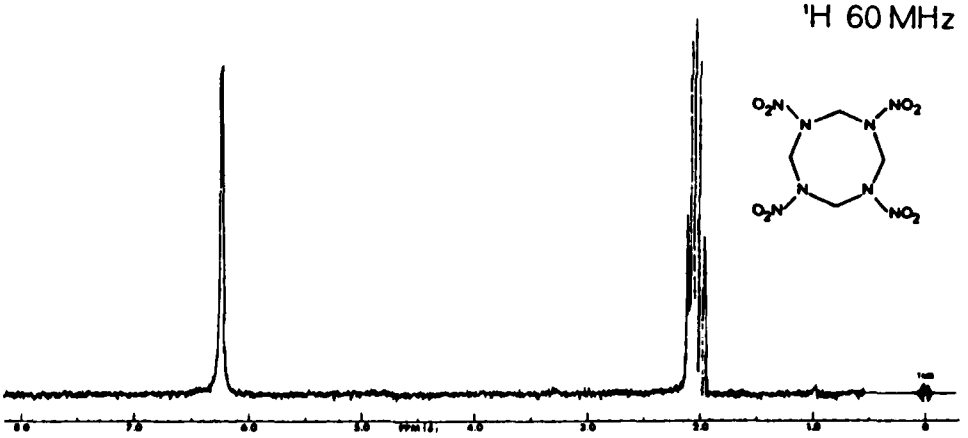


Group frequences (cm^{-1}):

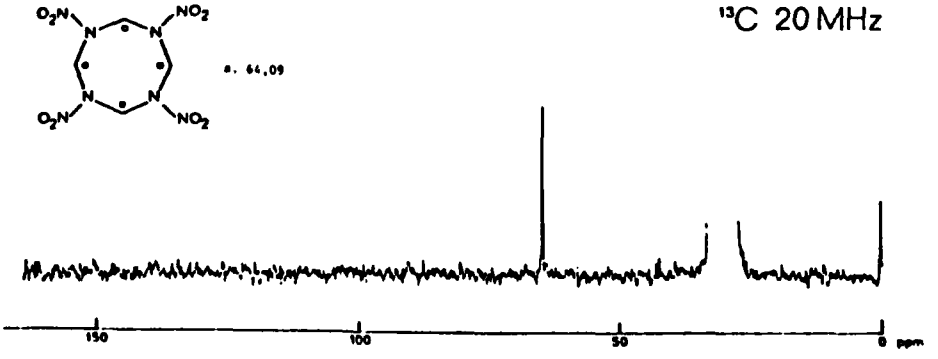
3042, 3032, 2986 C-H aliph — 1462, 1395 C-H aliph — 1147, 968 Ringabsorption

1560, 1540, 1542 (N)-NO_2 asym — 1298, 1280 (N)-NO_2 sym

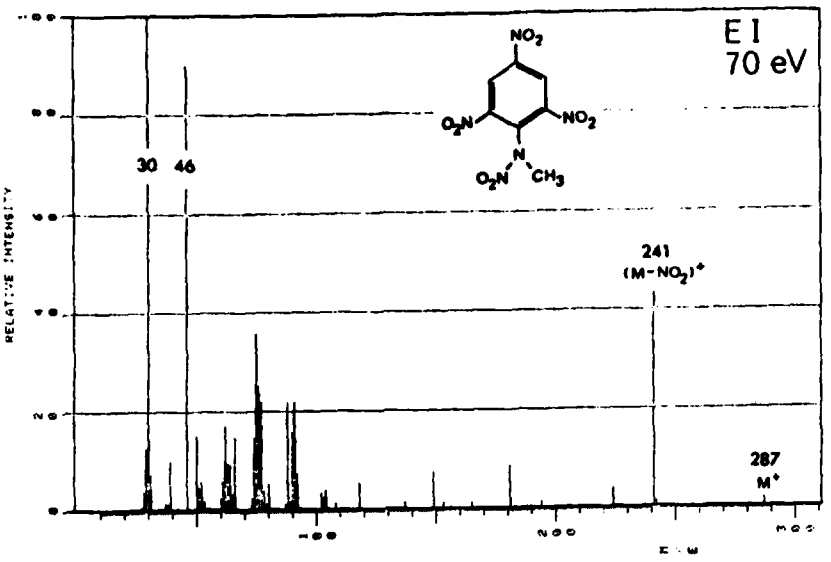
^1H 60 MHz



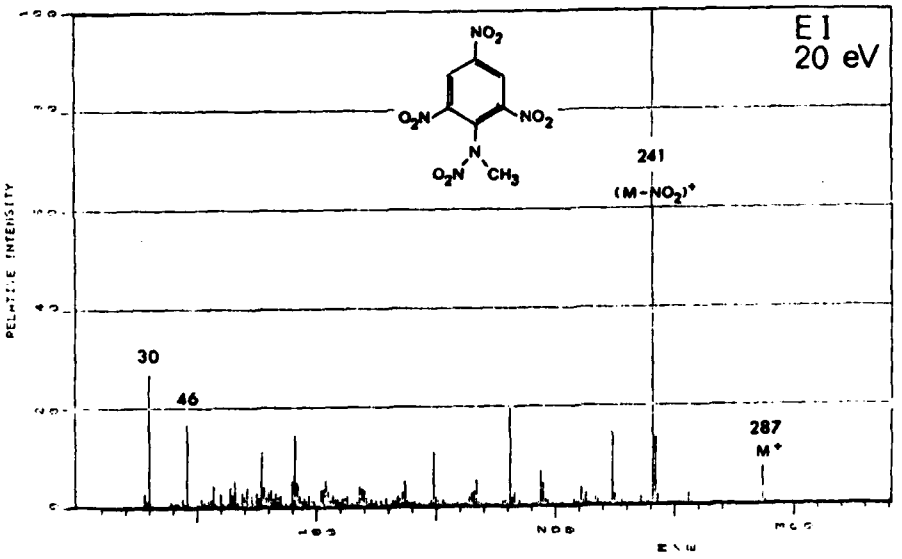
^{13}C 20 MHz



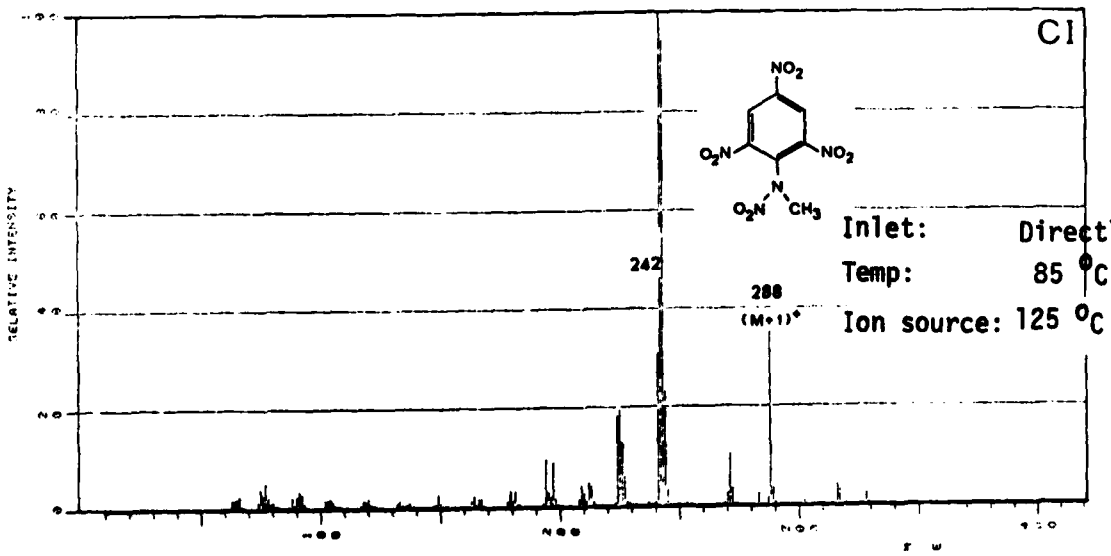
TETR



Inlet: Directly
Temp: 85 °C
Ion source: 150 °C

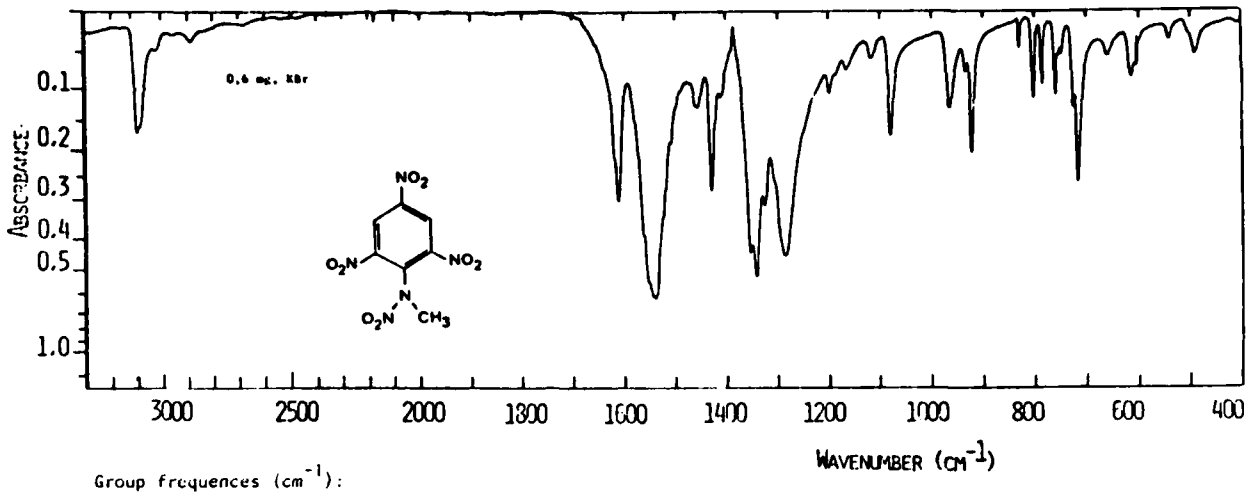


Inlet: Directly
Temp: 85 °C
Ion source: 150 °C



Inlet: Directly
Temp: 85 °C
Ion source: 125 °C

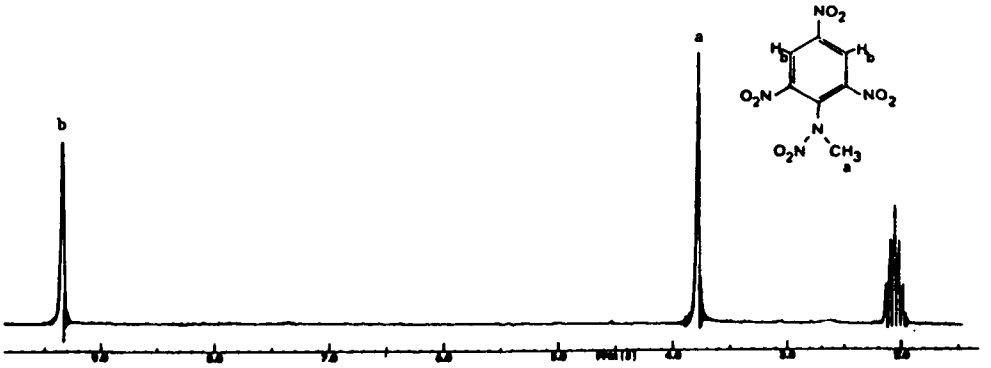
(7)



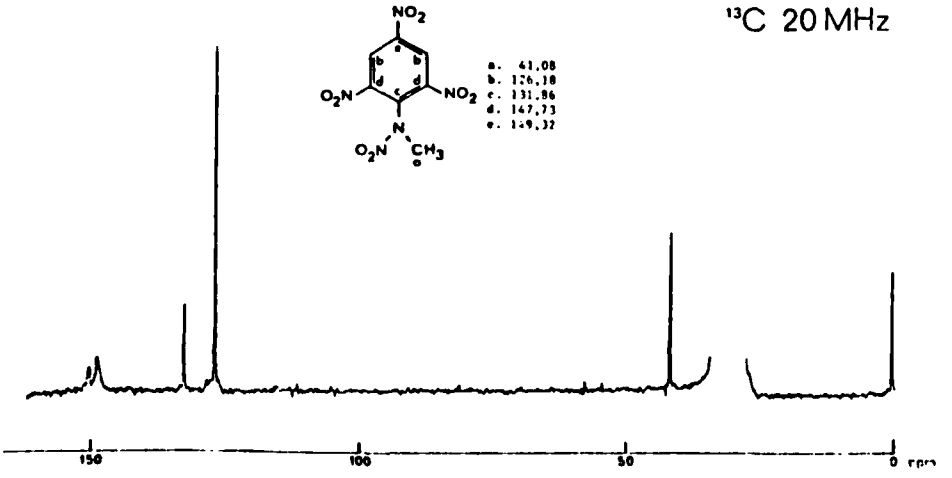
Group frequences (cm^{-1}):

3100, 3084, 3022	C-H arom	1611	phenyl	1354, 1341	(C)-NO_2 sym
2958	C-H aliph	1552, 1545, 1539	(C)-NO_2 asym och (N)-NO_2	1287	(N)-NO_2 sym
				1080, 922	Isol C-H arom

^1H 60 MHz



^{13}C 20 MHz



AM-PIKR

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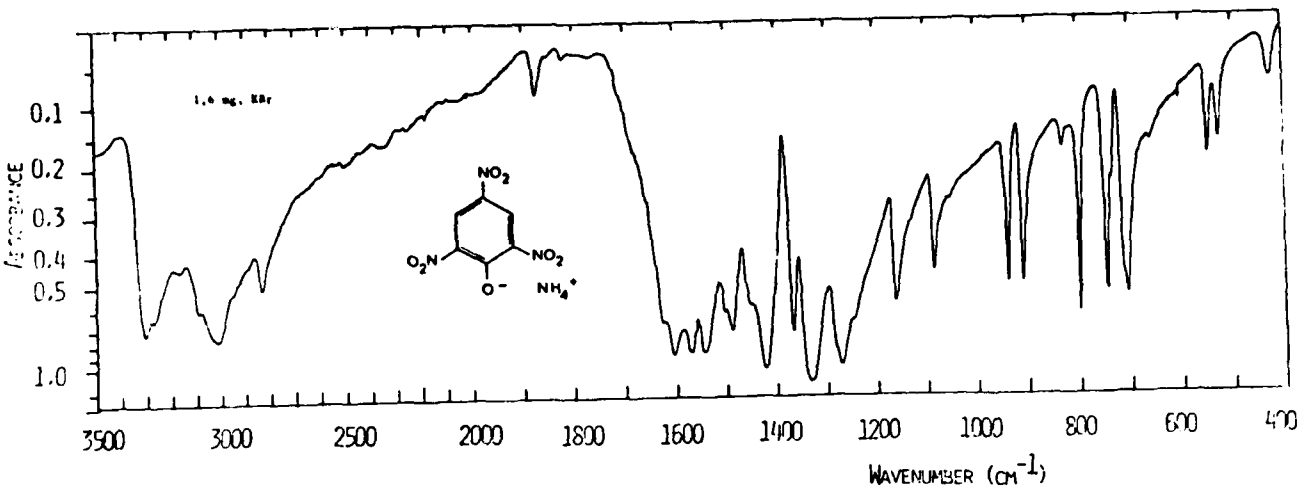
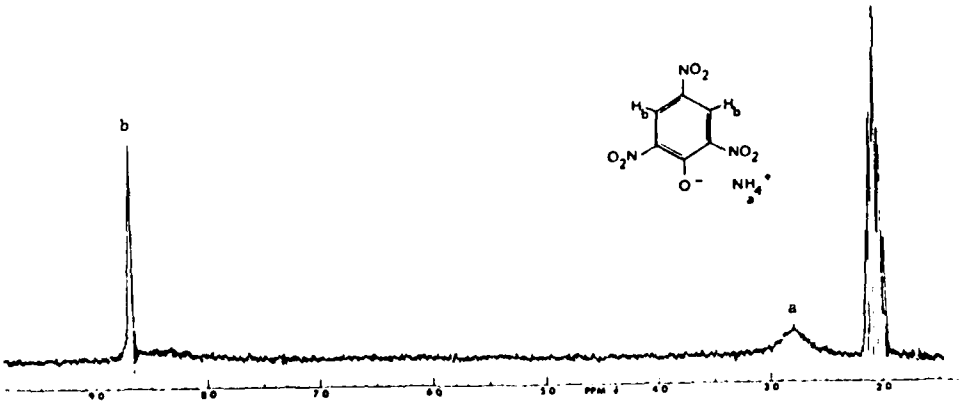
(8)



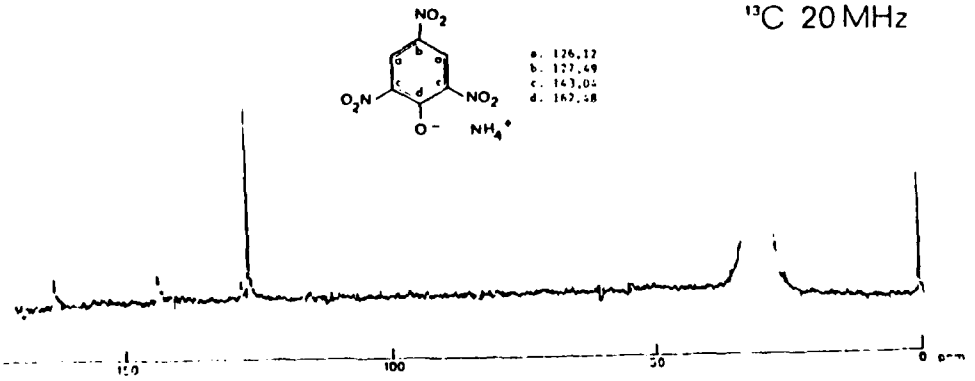
Group frequences (cm^{-1}):

3300, 3250	NH_4^+	3020	NH_4^+	1570, 1545	(C)-NO_2 asym	1333	(C)-NO_2 sym
3100	C-H arom	1624, 1604	phenyl	1422	NH_4^+	1080, 910	Isol C-H arom

^1H 60 MHz



^{13}C 20 MHz



HNS

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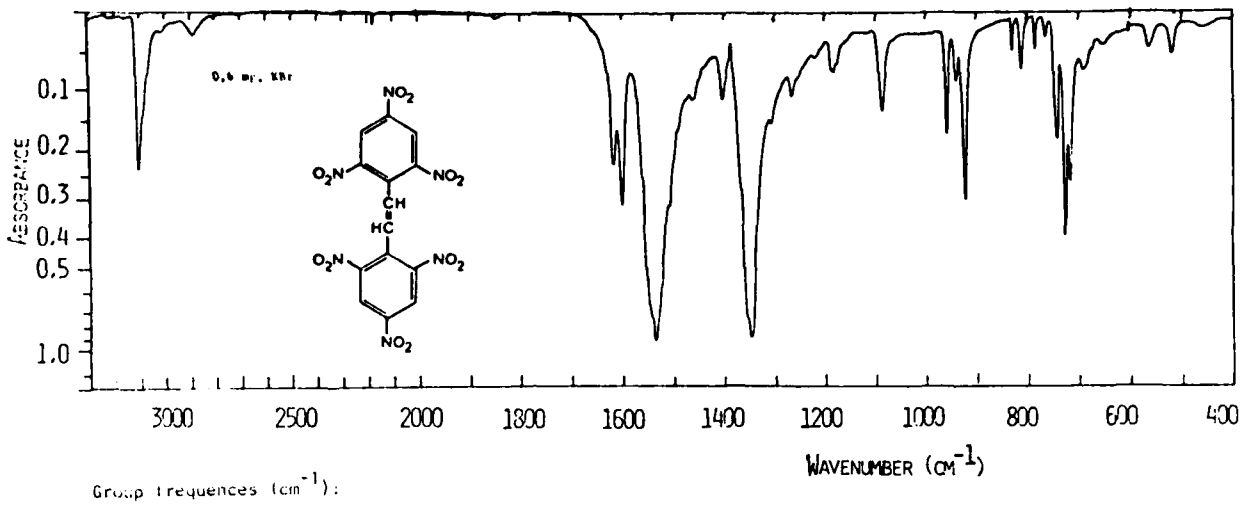
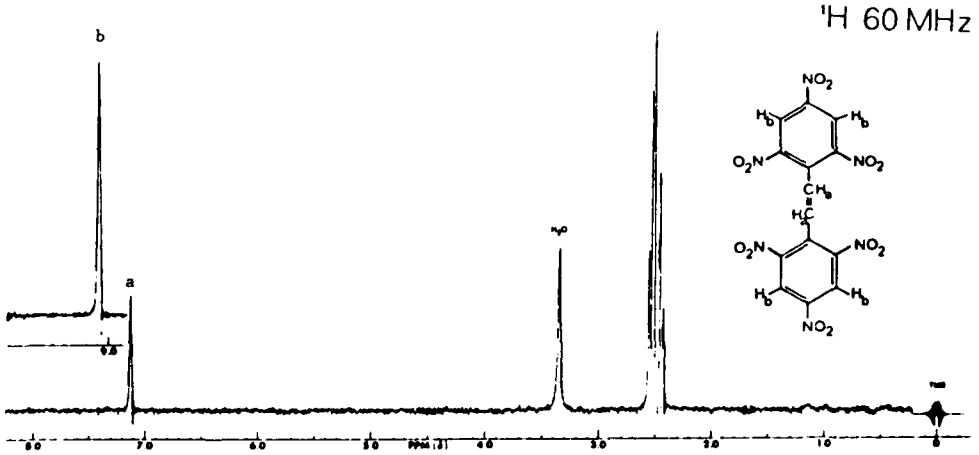


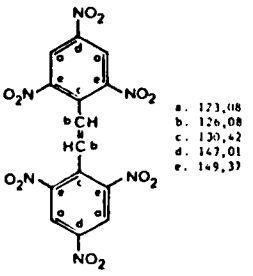
Group frequences (cm^{-1}):

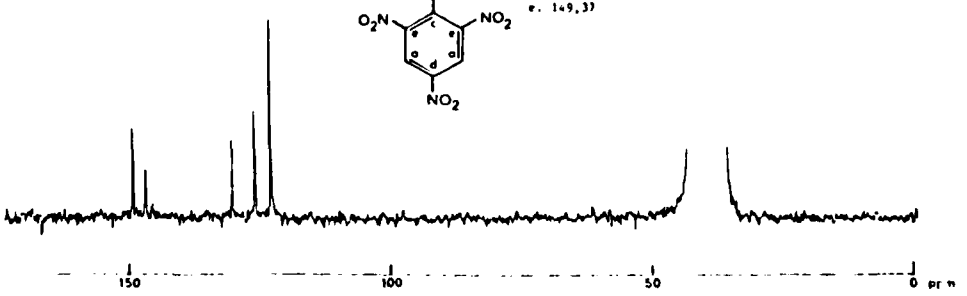
3105, 3083, 3074	C-H arom	1543, 1537	(C)-NO_2 asym	960	Transalken
1618, 1601	phenyl	1353, 1349	(C)-NO_2 sym	1087, 922	C-H arom

^1H 60 MHz

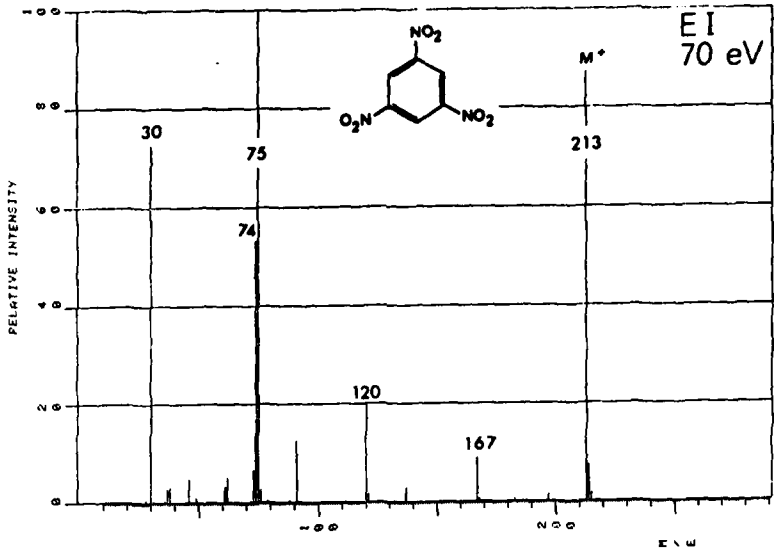


^{13}C 20 MHz

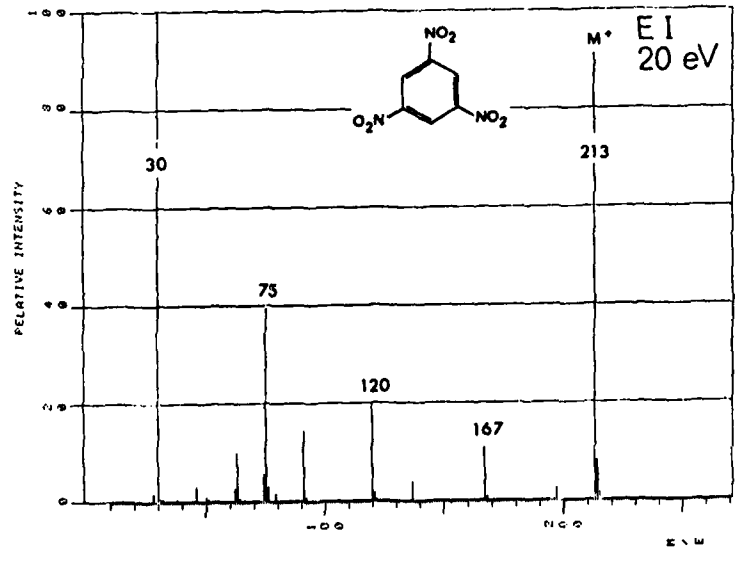




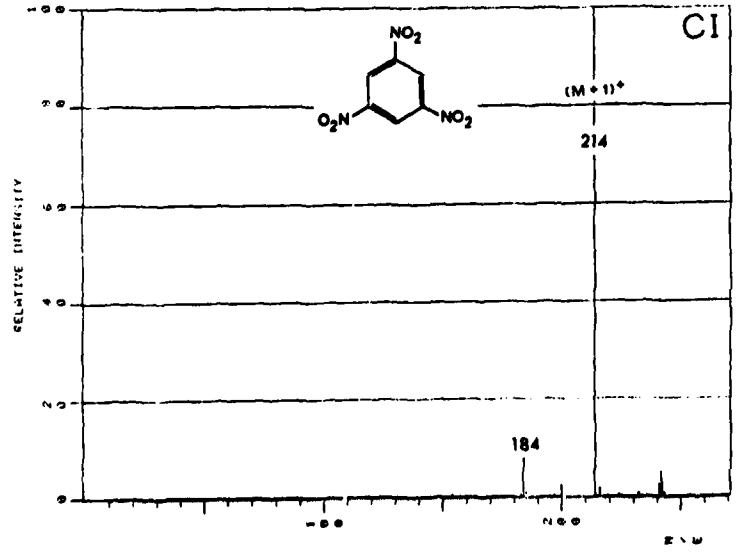
TNB ; 1,3,5-TNB



Inlet: GC
Column: OV 225
Ion source: 150 °C

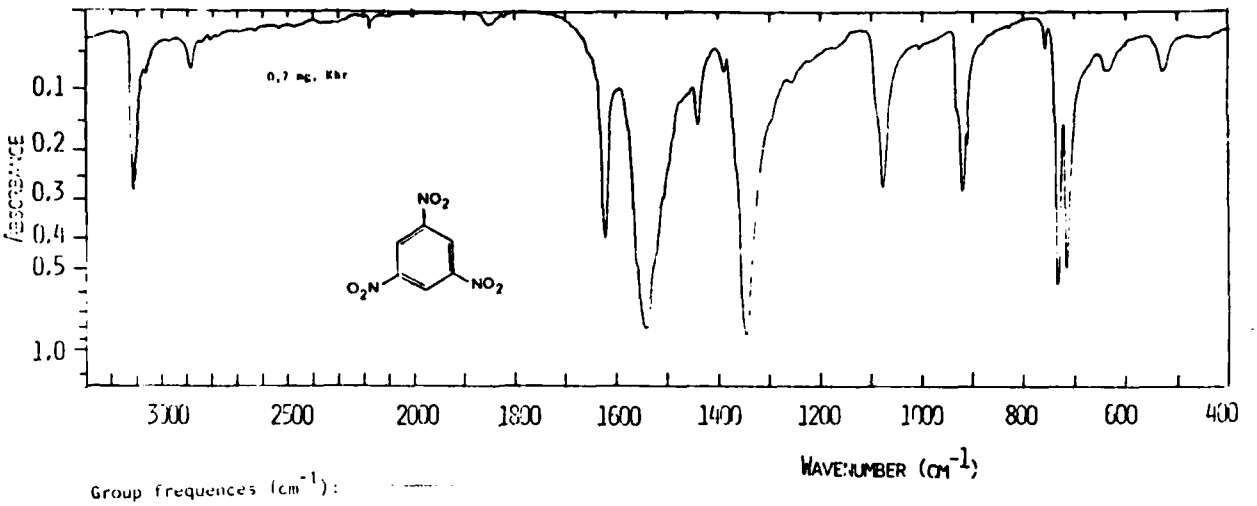


Inlet: GC
Column: OV 225
Ion source: 150 °C



Inlet: GC
Column: OV 225
Ion source: 150 °C

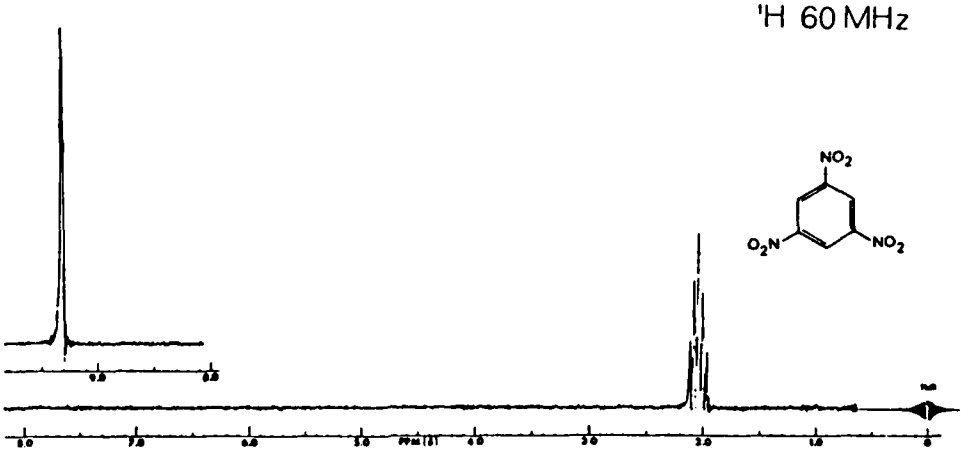
(10)



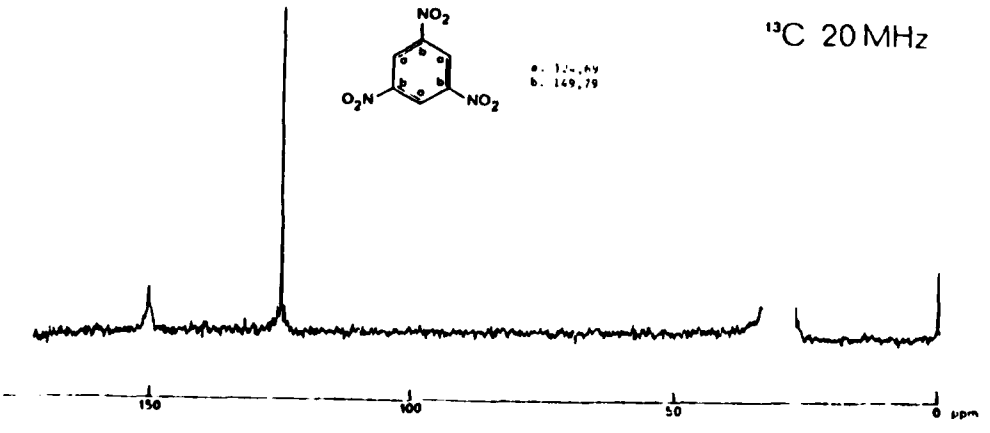
Group frequences (cm^{-1}):

3115, 3052 C-H arom	1542 (C)-NO_2 asym	1080, 922 Isol C-H arom
1622, 1440 phenyl	1347 (C)-NO_2 sym	

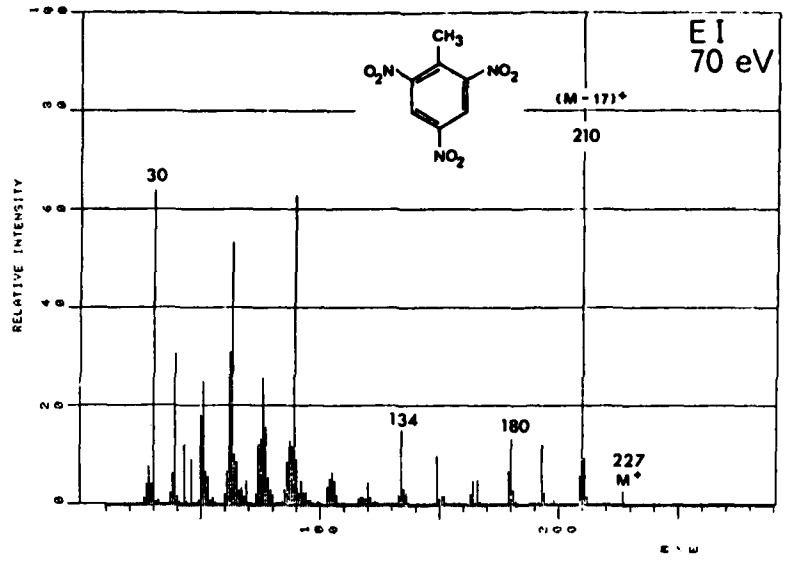
^1H 60 MHz



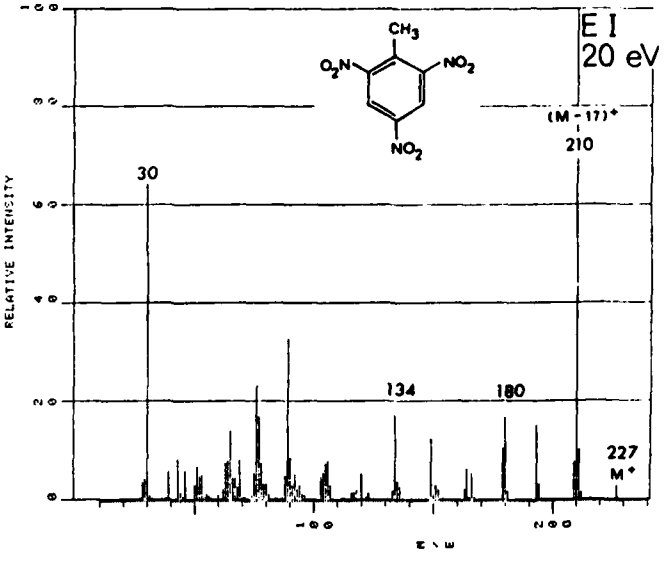
^{13}C 20 MHz



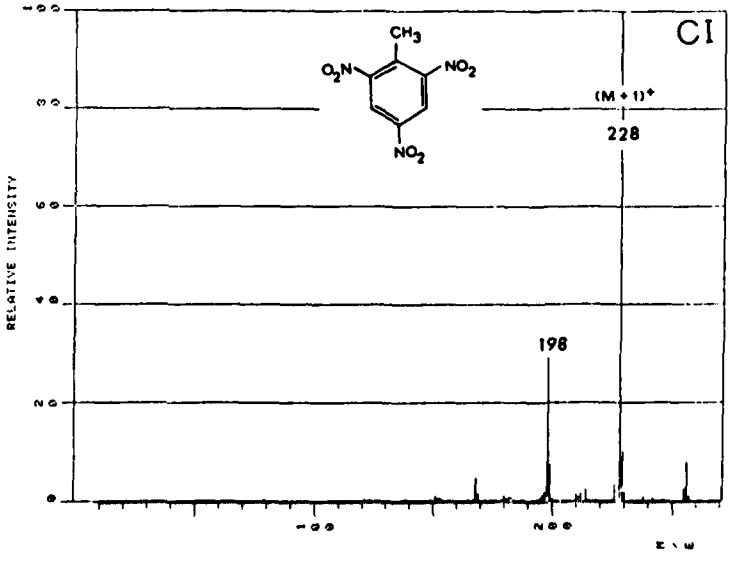
TNT ; 2,4,6 -TNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

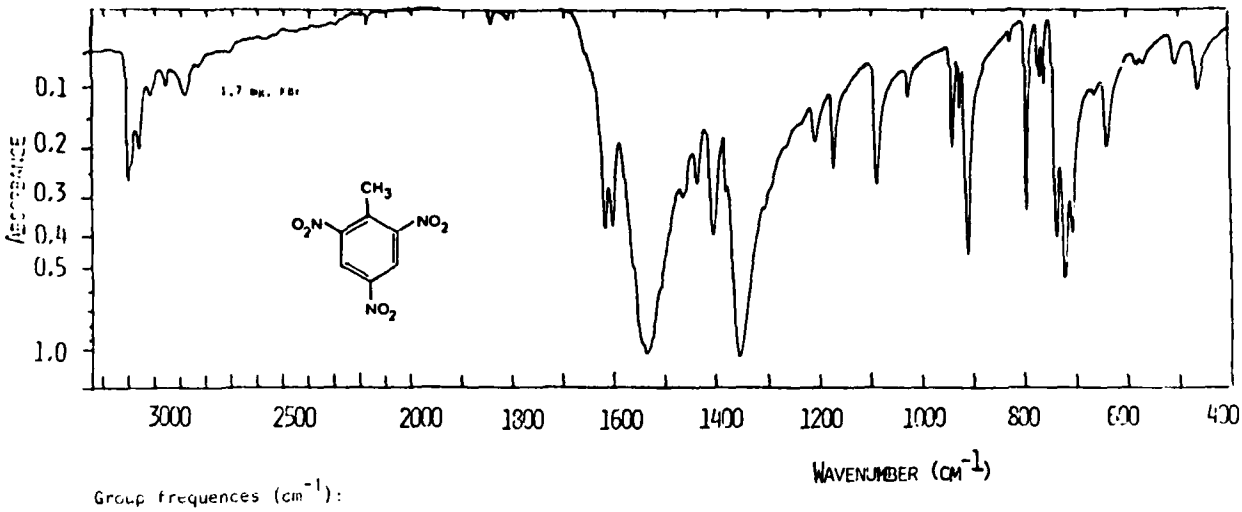


Inlet: GC
Column: OV 225
Ion source: 150 °C



Inlet: GC
Column: OV 225
Ion source: 150 °C

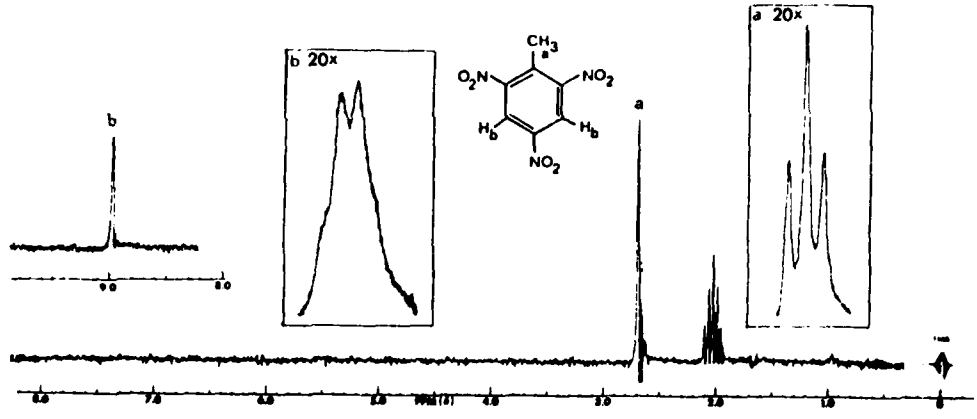
(11)



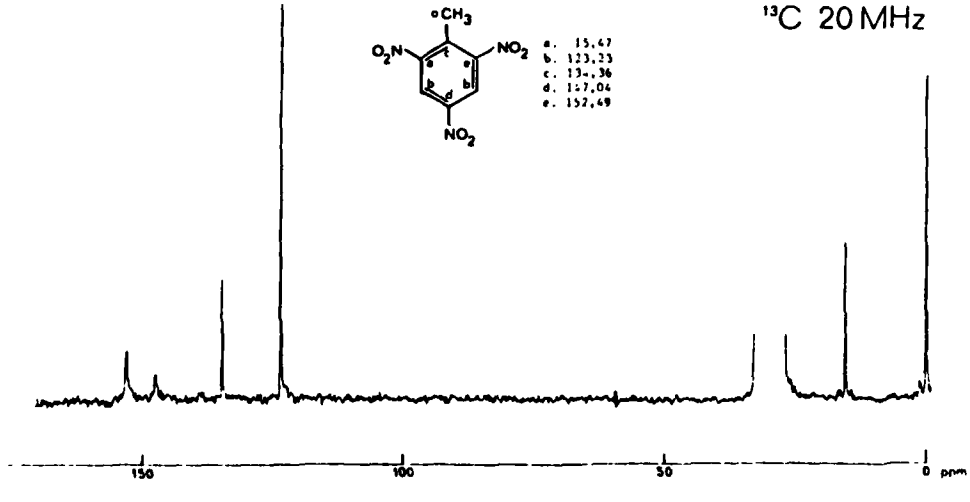
Group frequences (cm^{-1}):

3104, 3060, 3021	C-H arom	1622, 1604	phenyl	1358	(C)-NO_2 sym
2960	C-H aliph	1544, 1536	(C)-NO_2 asym	1090, 910	Isol C-H arom

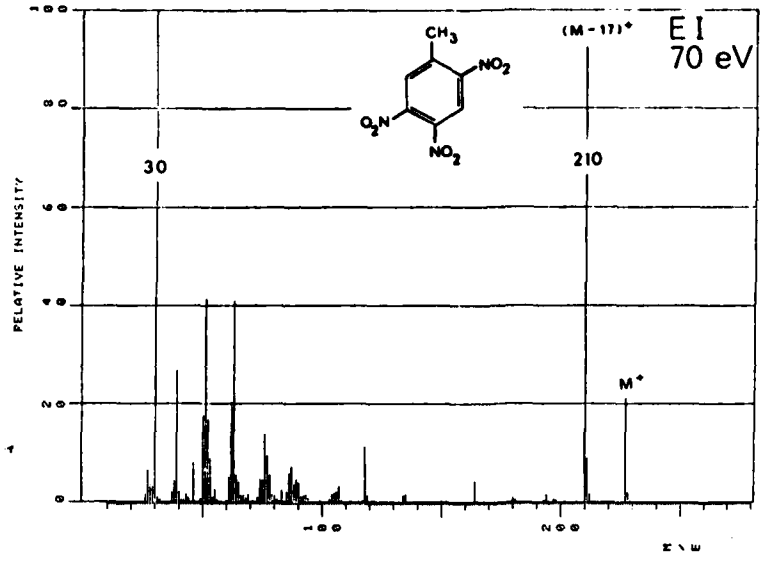
^1H 60 MHz



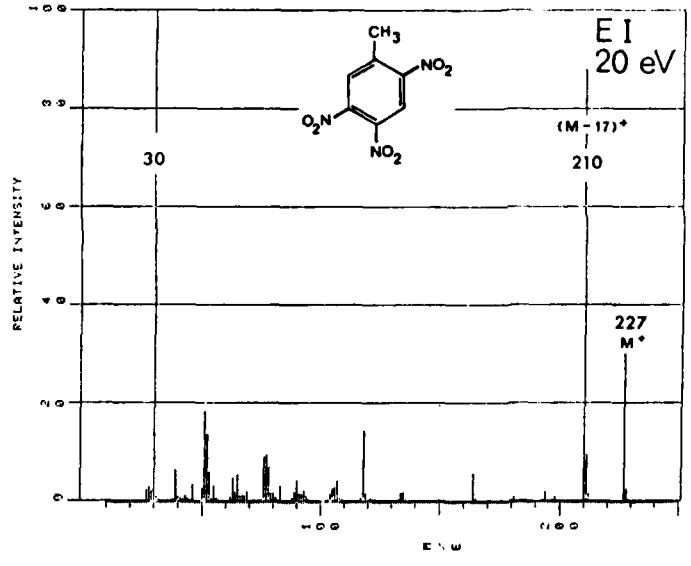
^{13}C 20 MHz



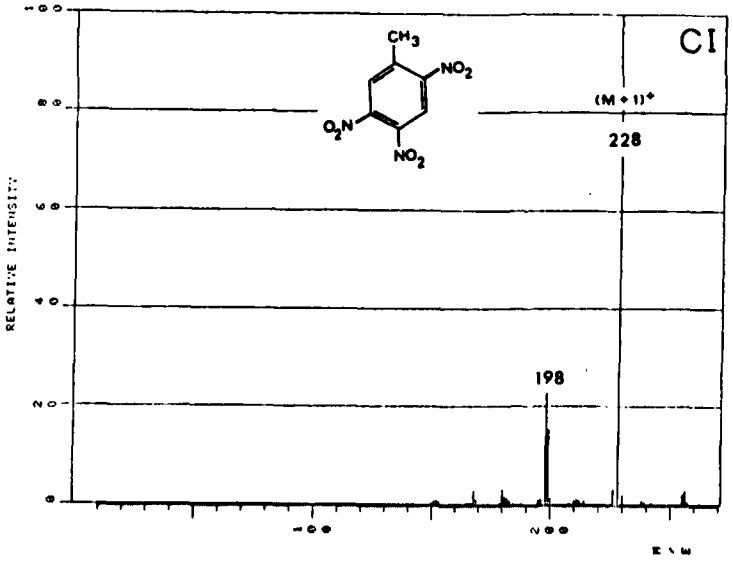
2,4,5 - TNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

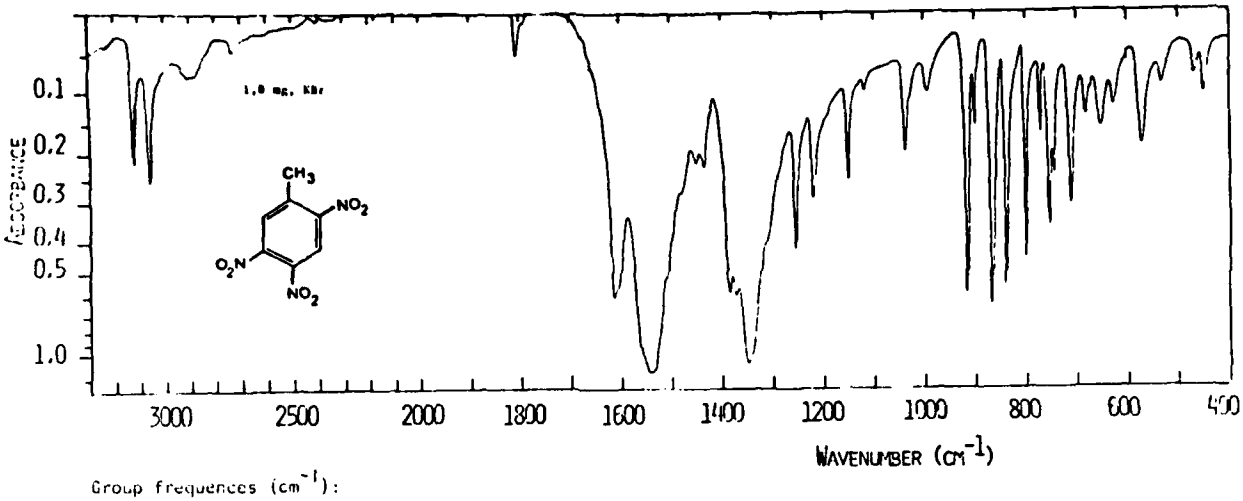


Inlet: GC
Column: OV 225
Ion source: 150 °C



Inlet: GC
Column: OV 225
Ion source: 150 °C

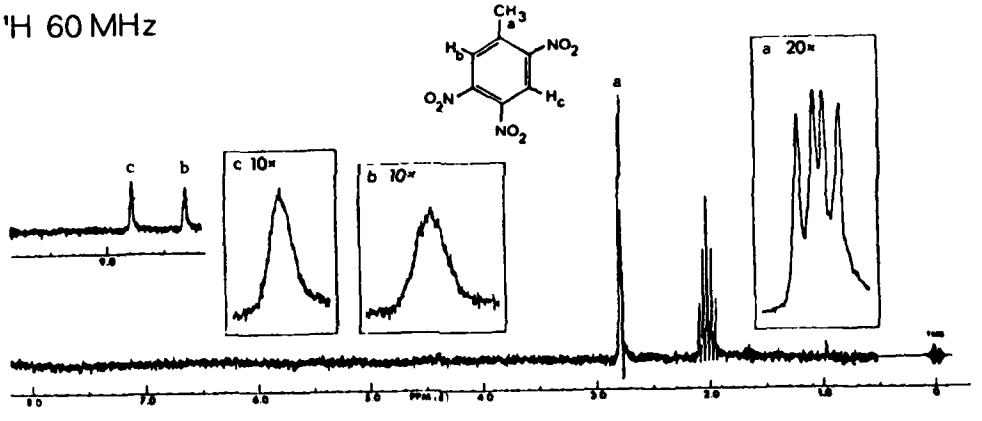
(12)



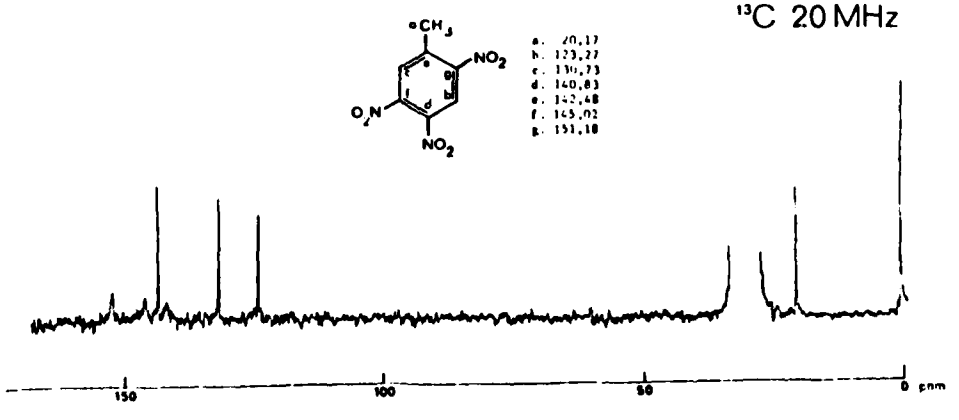
Group frequences (cm^{-1}):

3120, 3060	C-H arom	1563, 1547, 1537	(C)-NO_2 asym	1040, 918	Isol C-H arom
1612, 1604	phenyl	1350	(C)-NO_2 sym		

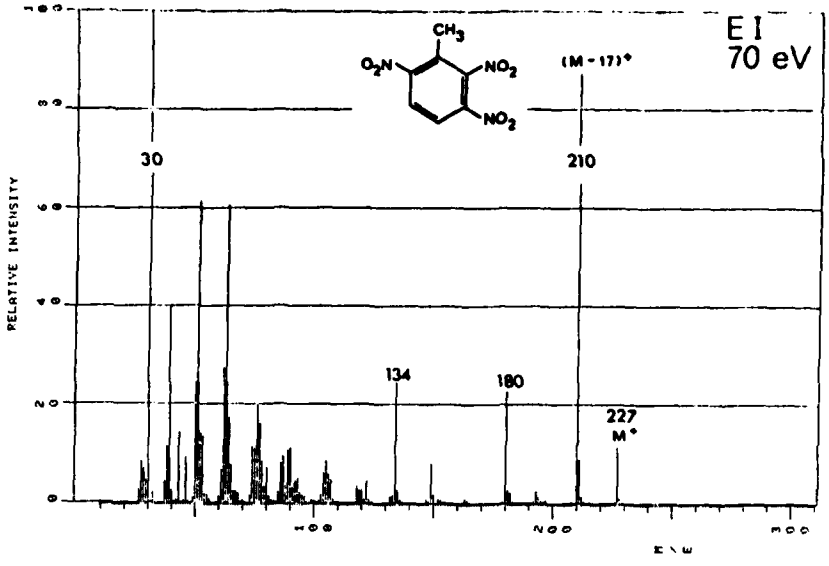
^1H 60 MHz



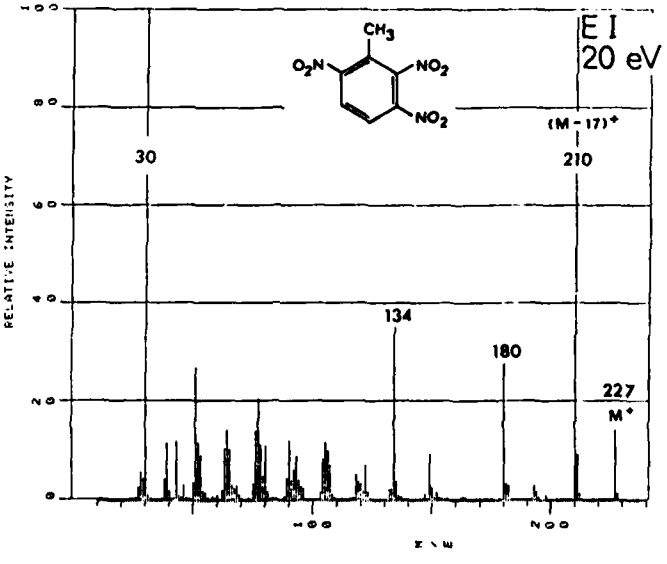
^{13}C 20 MHz



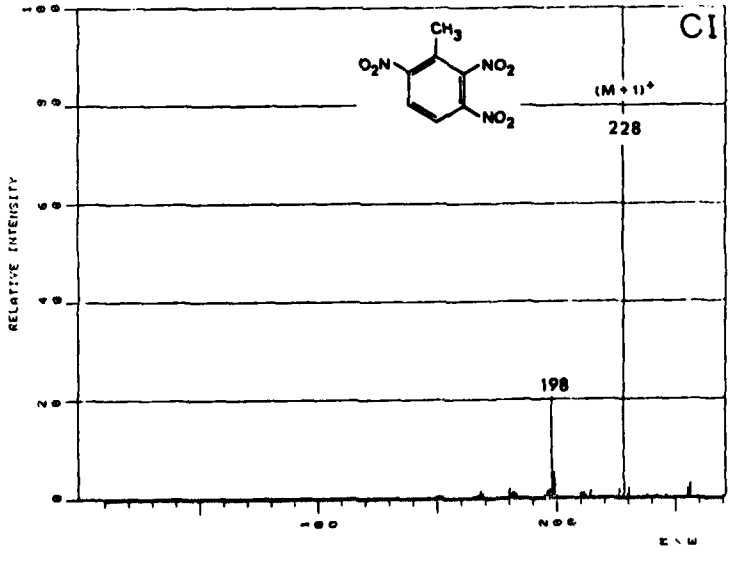
2,3,6 - TNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

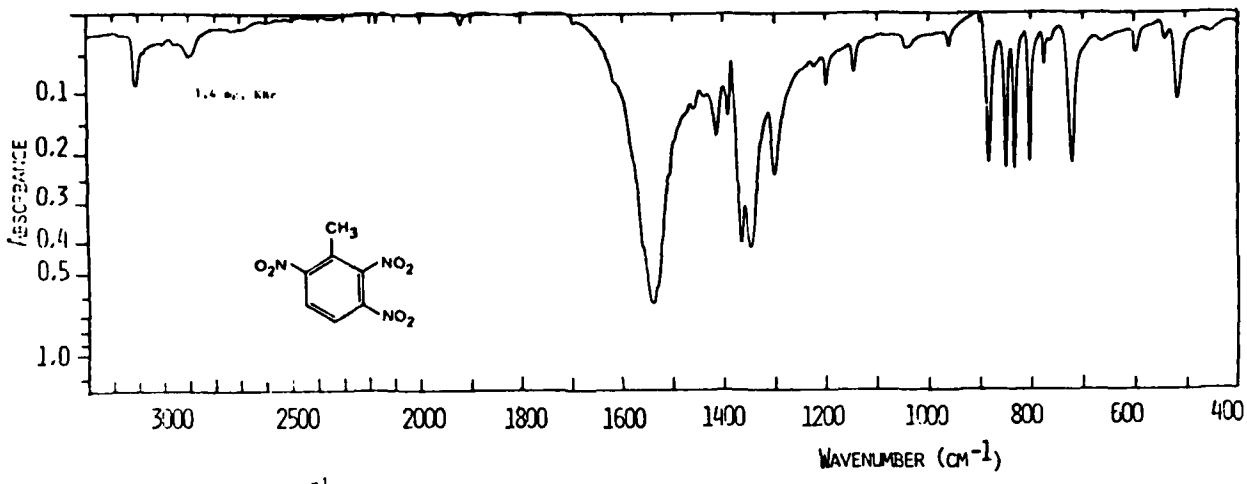


Inlet: GC
Column: OV 225
Ion source: 150 °C



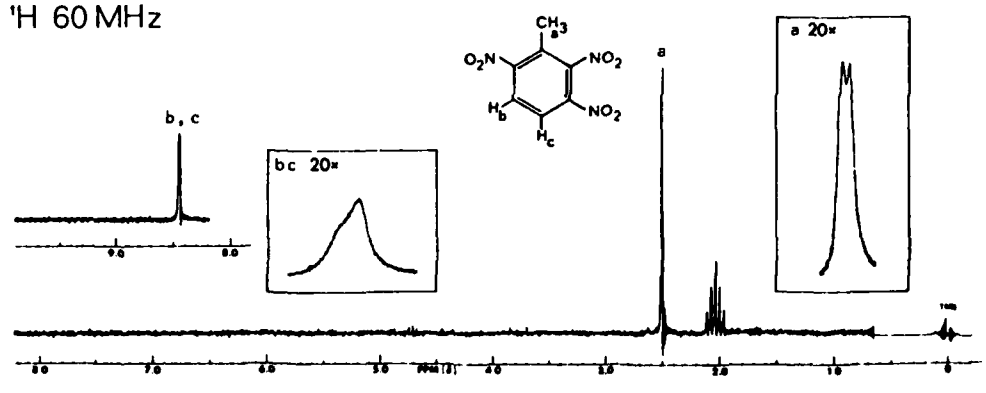
Inlet: GC
Column: OV 225
Ion source: 150 °C

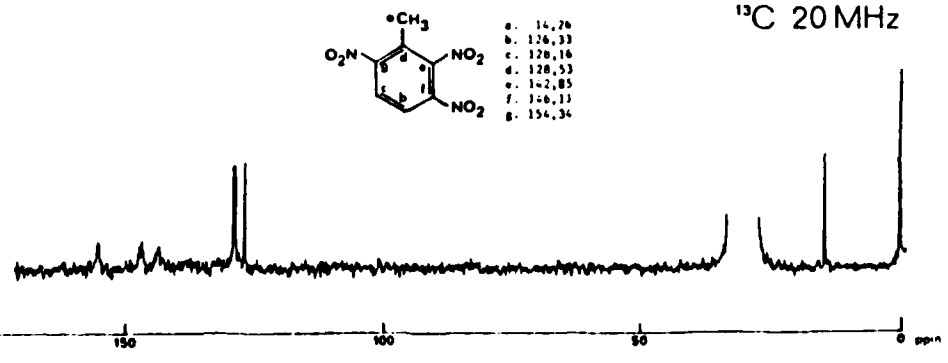
13



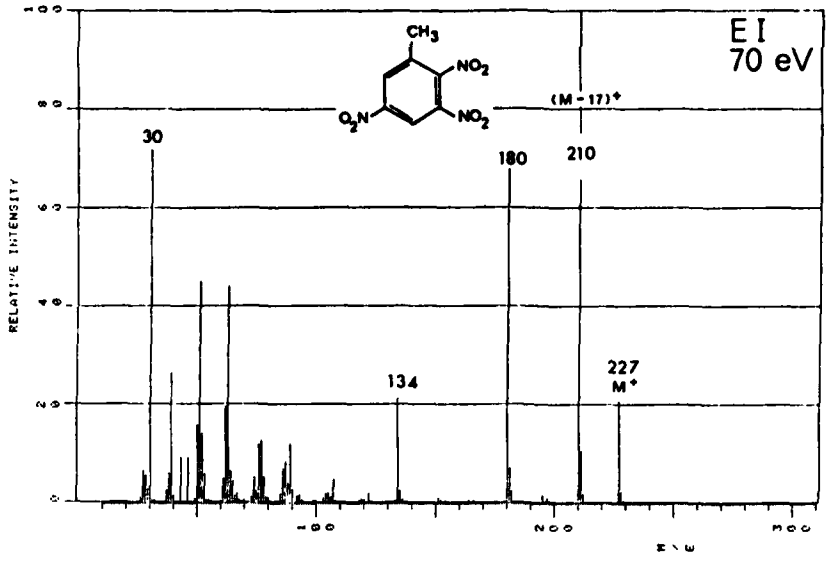
Group frequences (cm^{-1}):

3108	C-H arom	1367, 1345	(C)-NO_2 sym
1560, 1541, 1532	(C)-NO_2 asym		

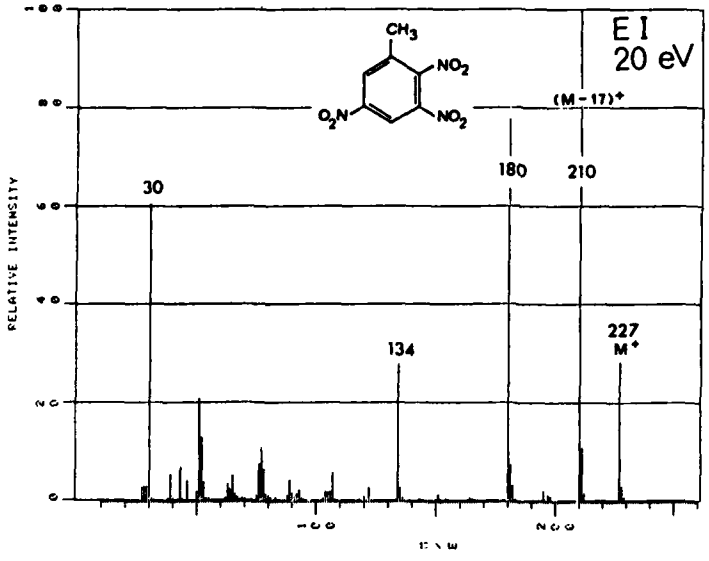




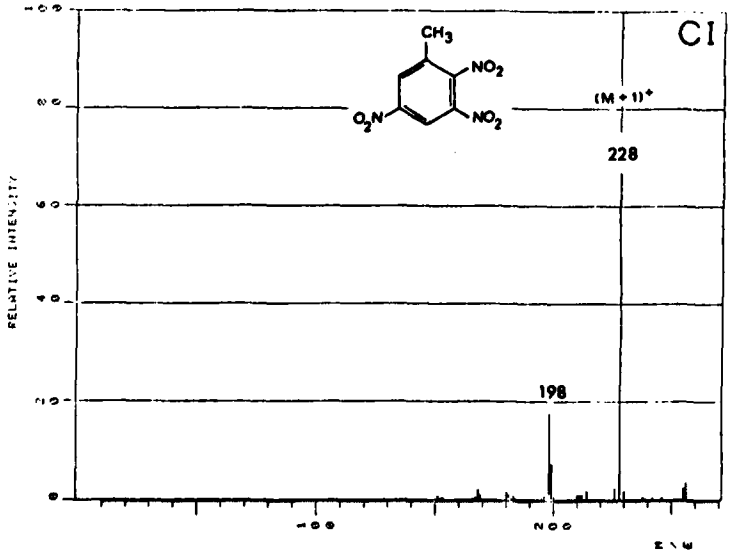
2,3,5 – TNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

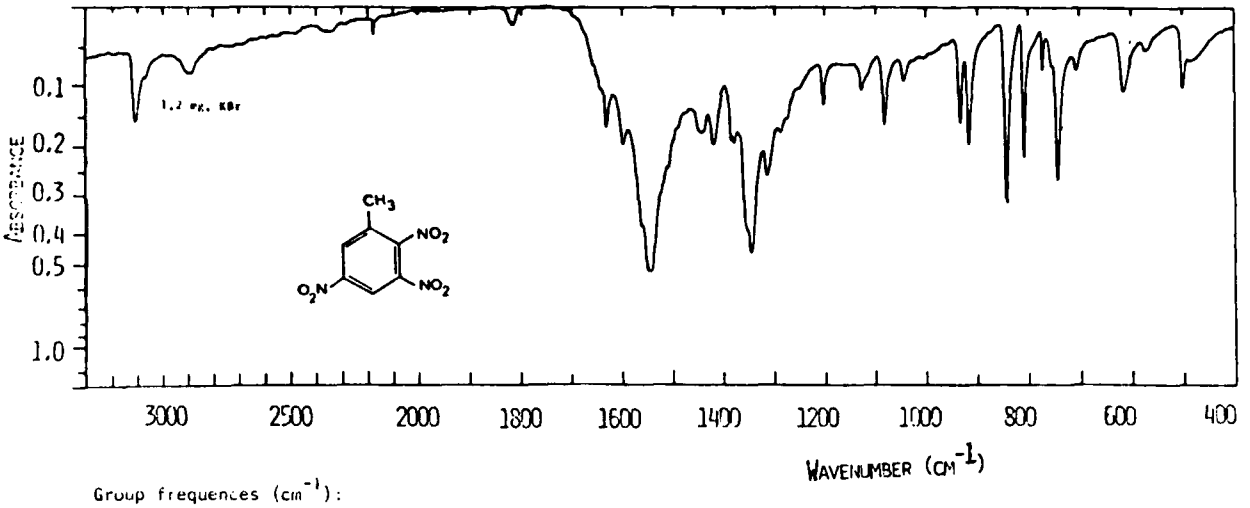


Inlet: GC
Column: OV 225
Ion source: 150 °C



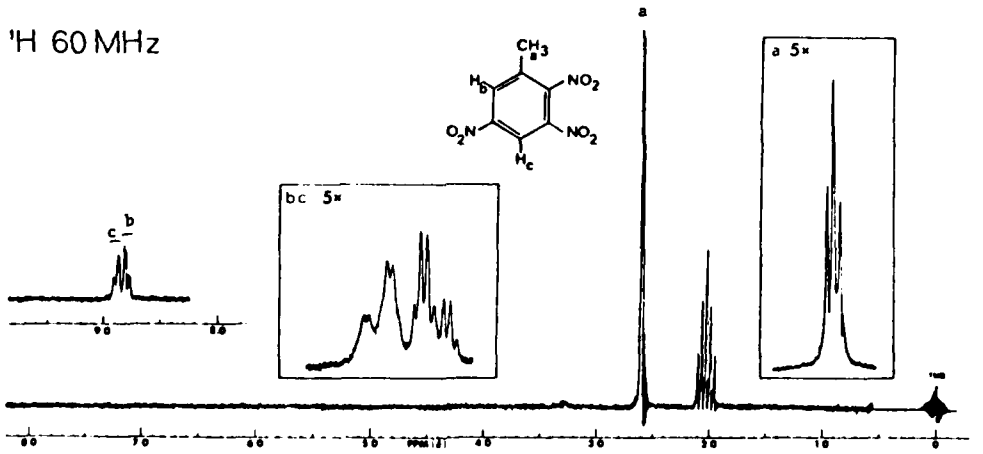
Inlet: GC
Column: OV 225
Ion source: 150 °C

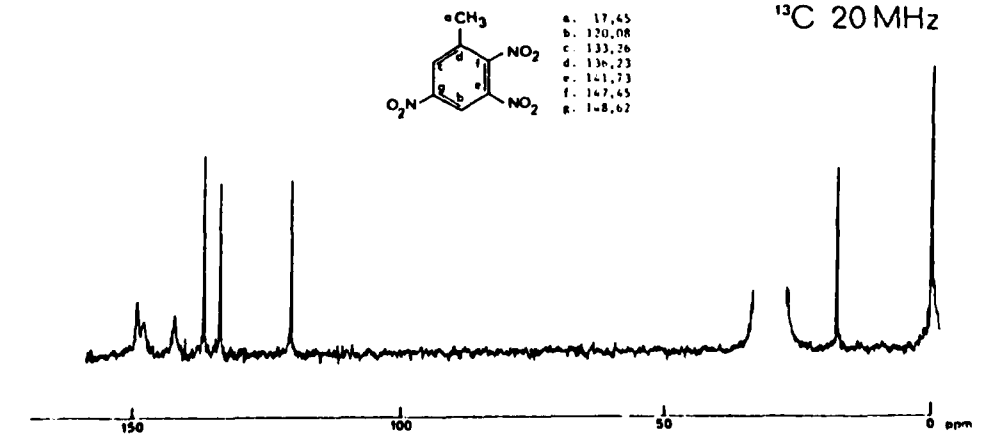
(14)



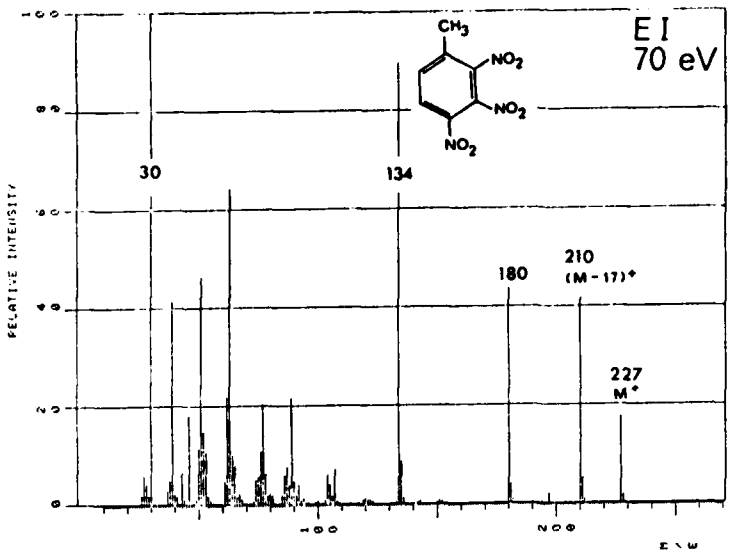
Group frequences (cm^{-1}):

3108, 3064 C-H arom	1563, 1545 (C)-NO_2 asym	1080, 919 Isol C-H arom	
1630, 1599 phenyl	1352, 1348 (C)-NO_2 sym		

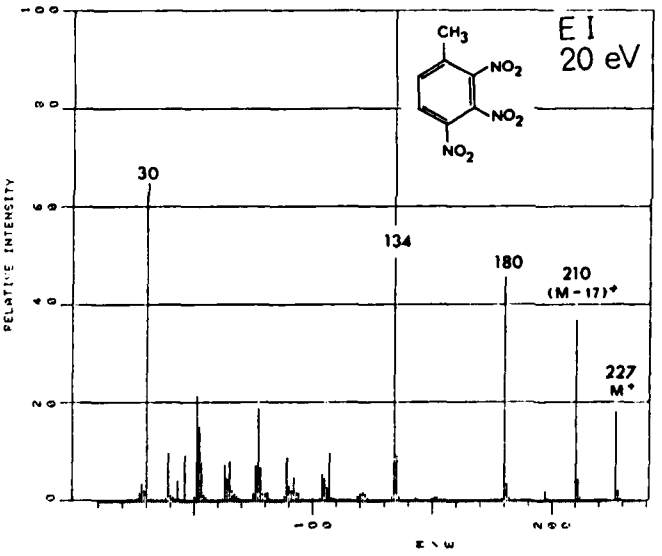




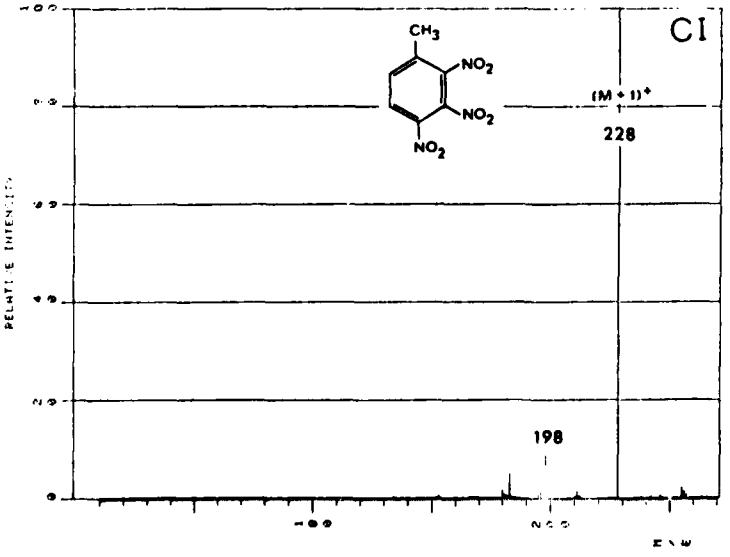
2,3,4 – TNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

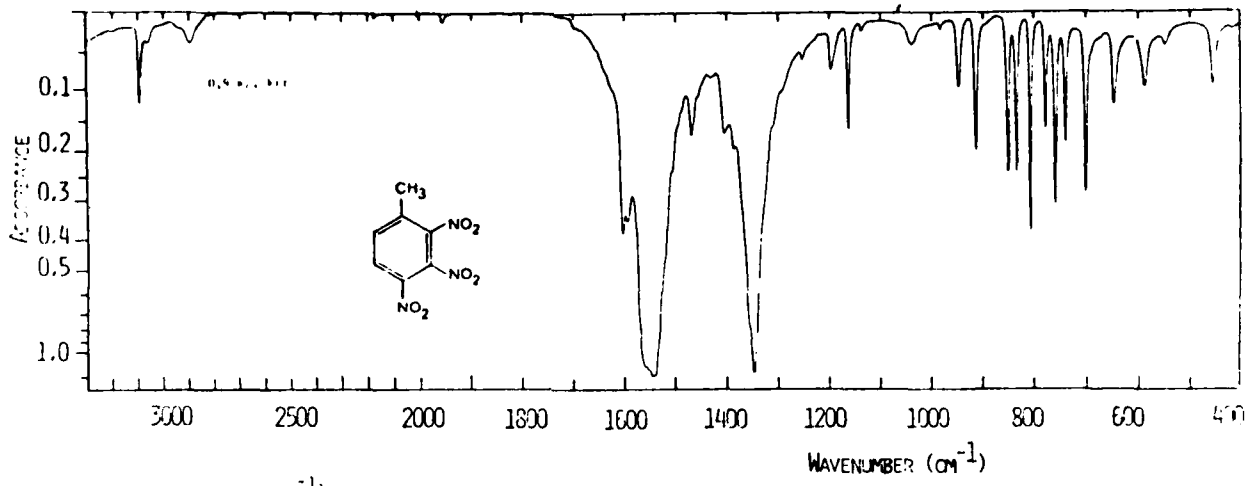


Inlet: GC
Column: OV 225
Ion source: 150 °C



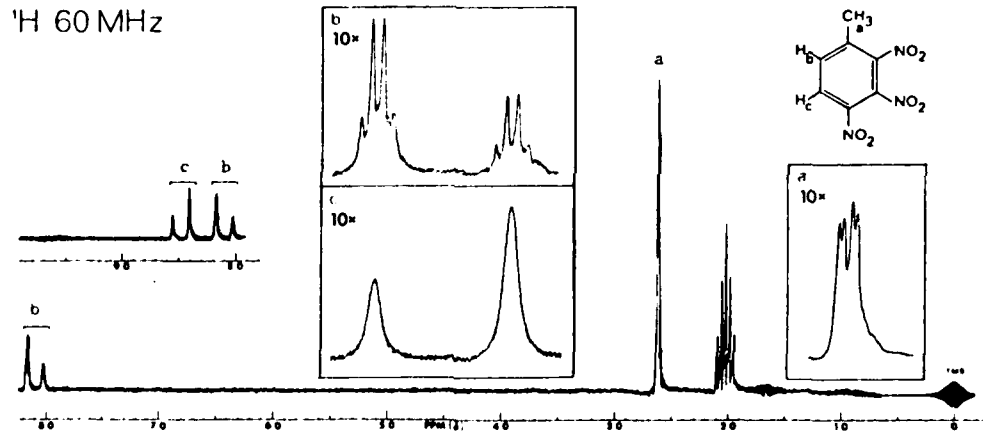
Inlet: GC
Column: OV 225
Ion source: 150 °C

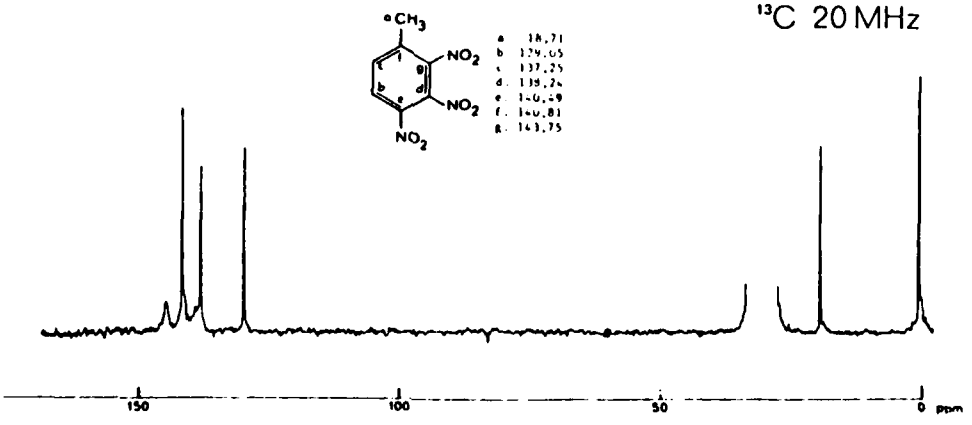
15



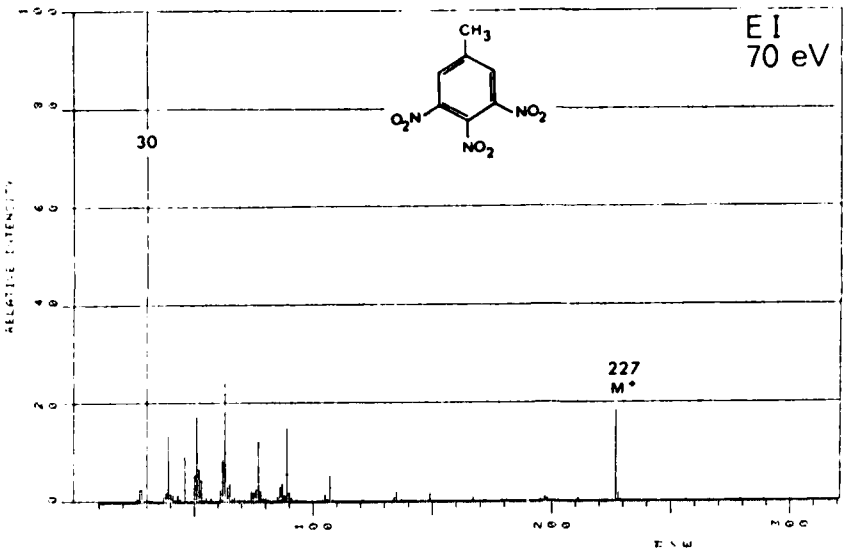
Group frequences (cm^{-1}):

3097, 3060	C-H aroa	1560, 1553, 1548	(C)-NO_2 asym
1608, 1597	phenyl	[illegible], 1348	(C)-NO_2 sym

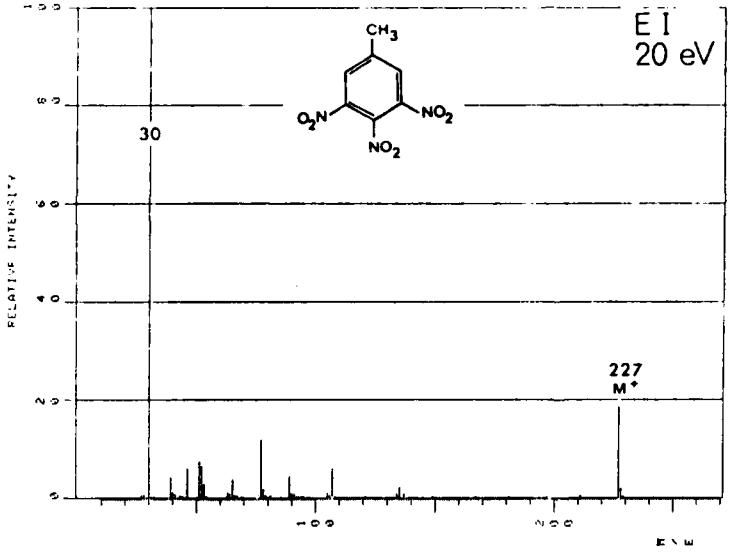




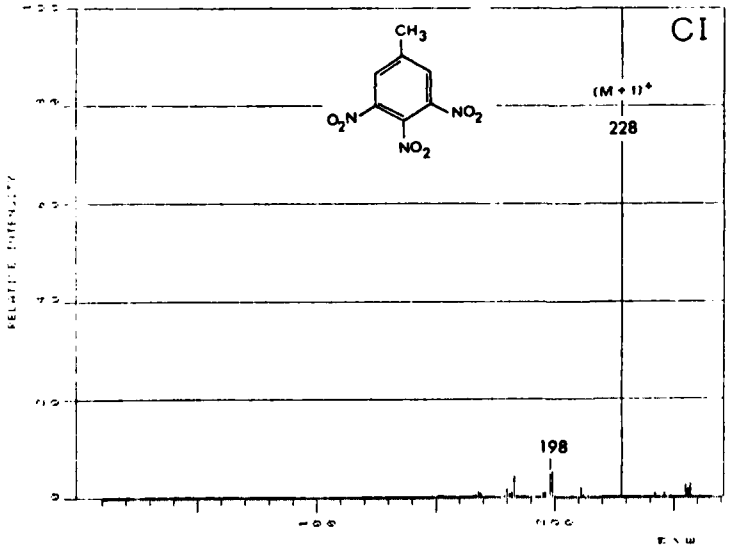
3,4,5 – TNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

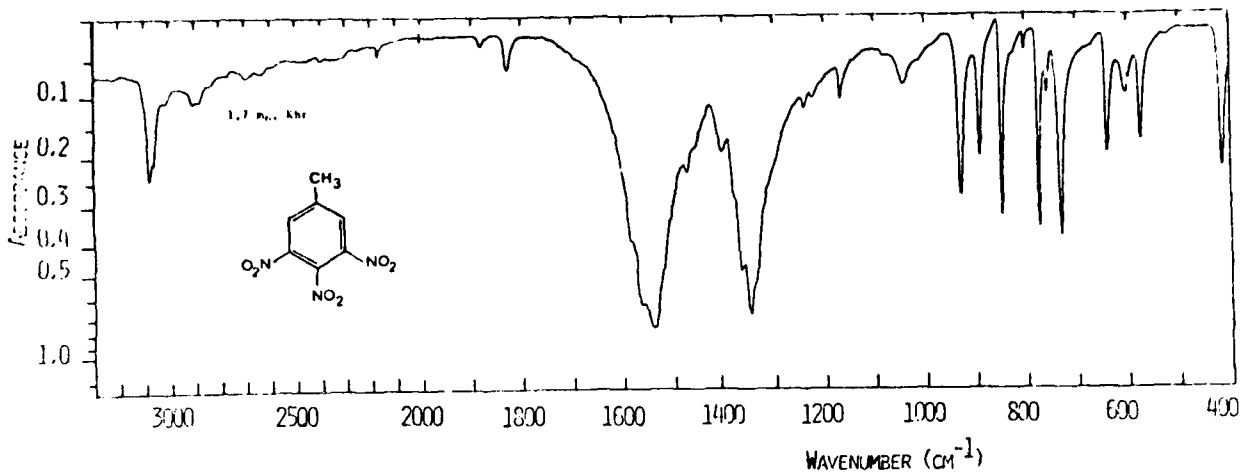


Inlet: GC
Column: OV 225
Ion source: 150 °C



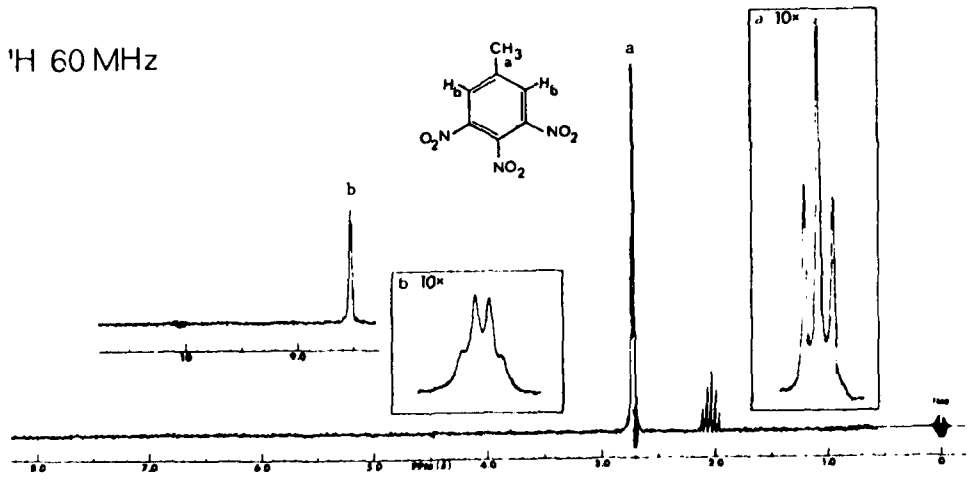
Inlet: GC
Column: OV 225
Ion source: 150 °C

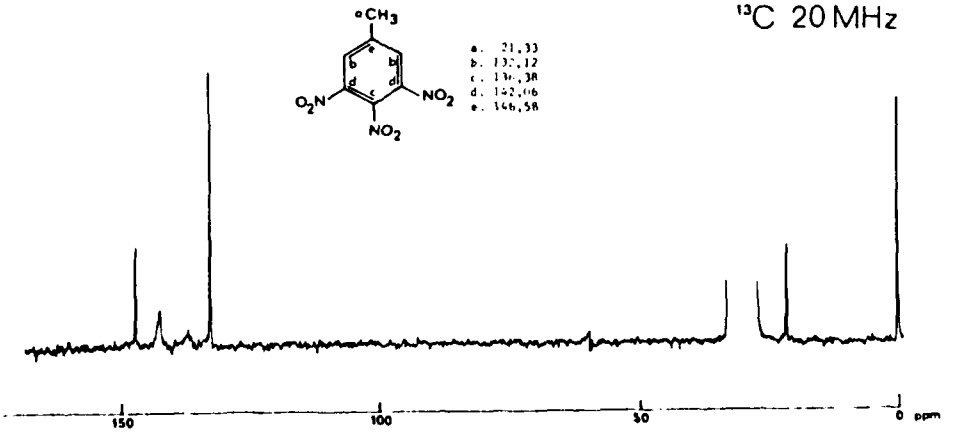
16



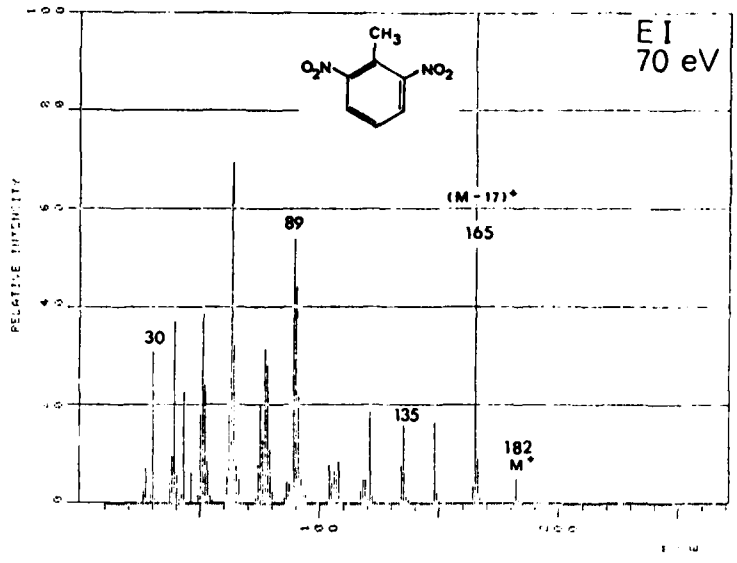
Group frequences (cm^{-1}):

3082, 3060	C-H arom	1370, 1350, 1342	(C)-NO_2 sym
1563, 1542	(C)-NO_2 asym	929	Isol C-H arom

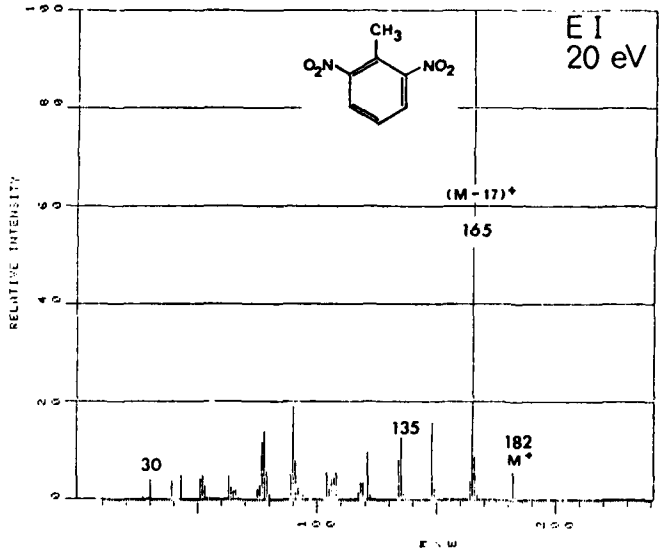




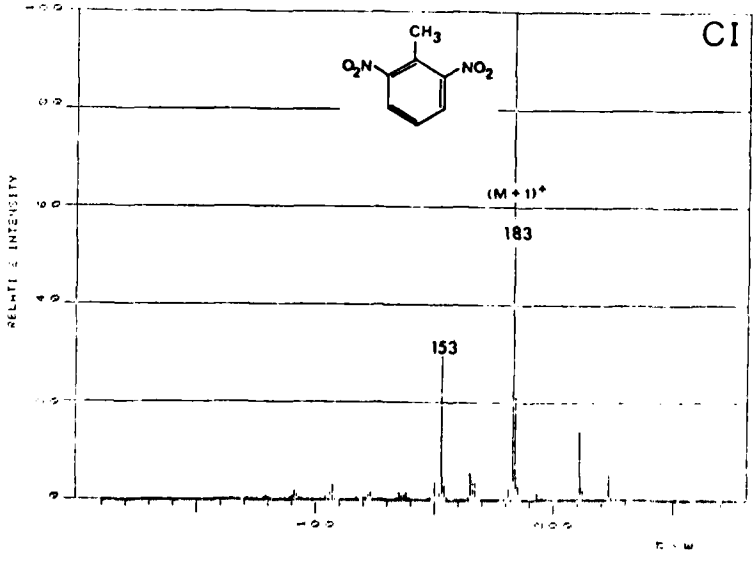
2,6 – DNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

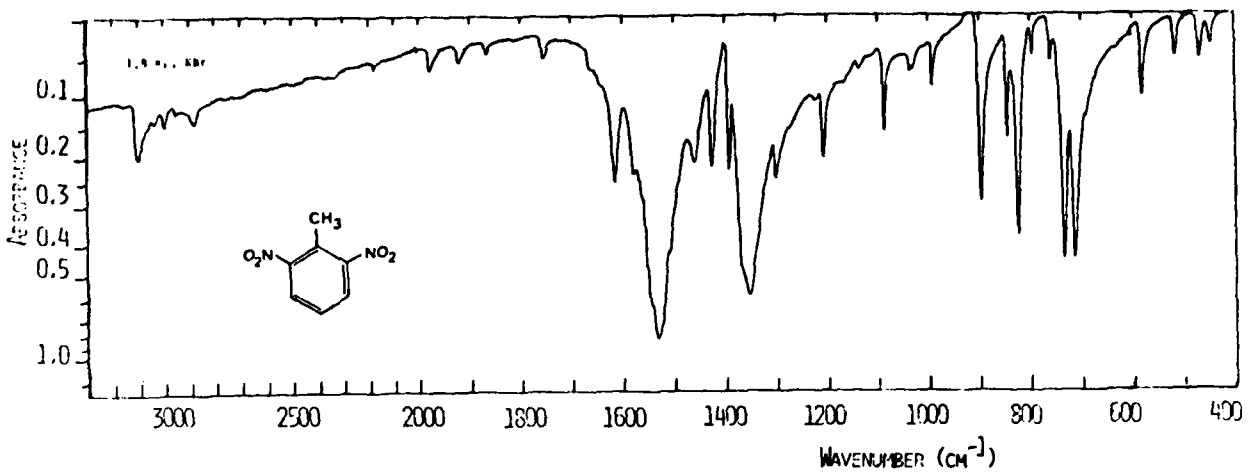


Inlet: GC
Column: OV 225
Ion source: 150 °C



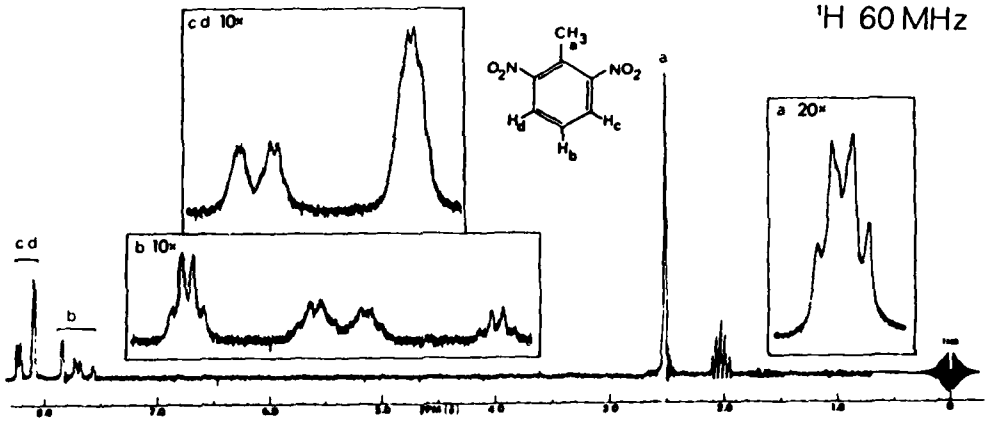
Inlet: GC
Column: OV 225
Ion source: 150 °C

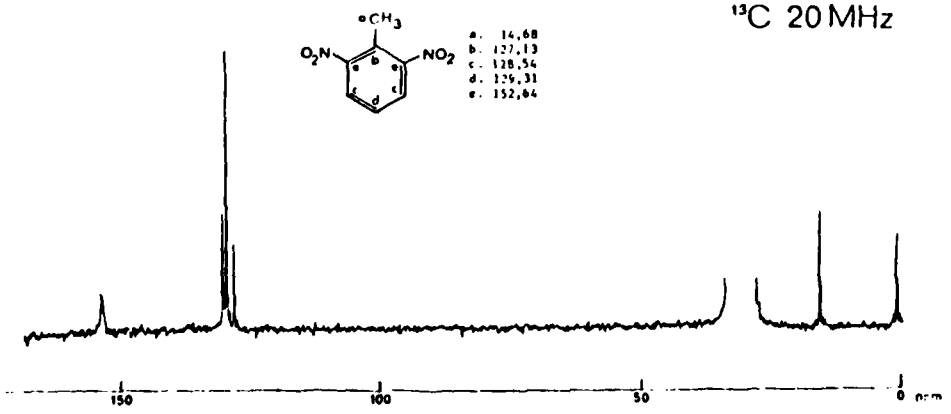
(17)



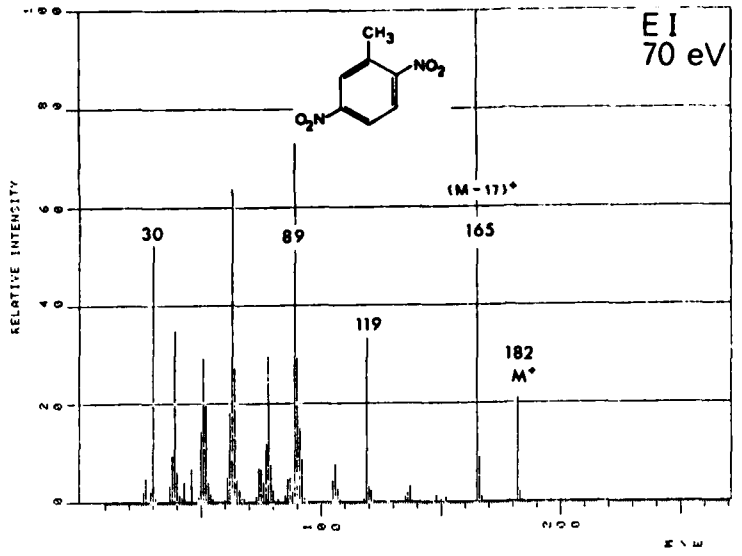
Group frequences (cm^{-1}):

3108, 3090, 3038	C-H arom	1614	phenyl	1365, 1352 (C)-NO_2 sym
3001, 2957	C-H aliph	1542, 1530, 1527	(C)-NO_2 asym	

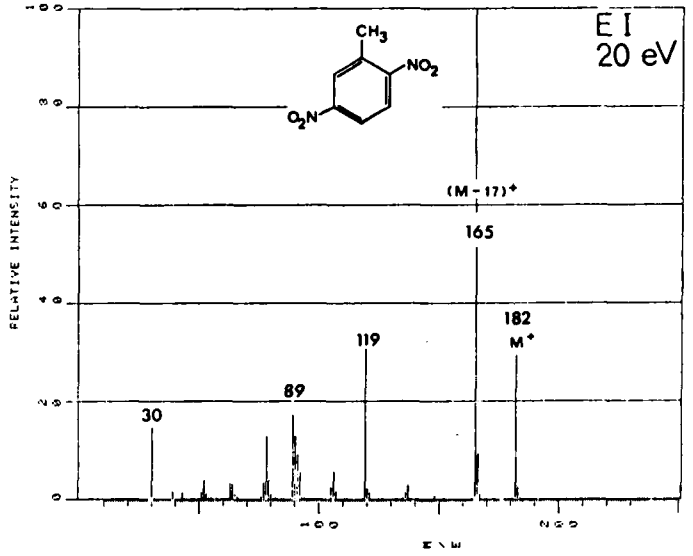




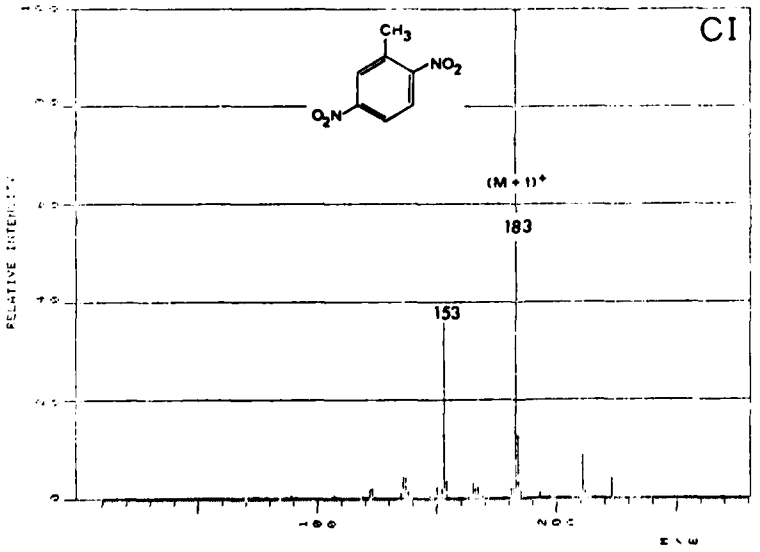
2,5 – DNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

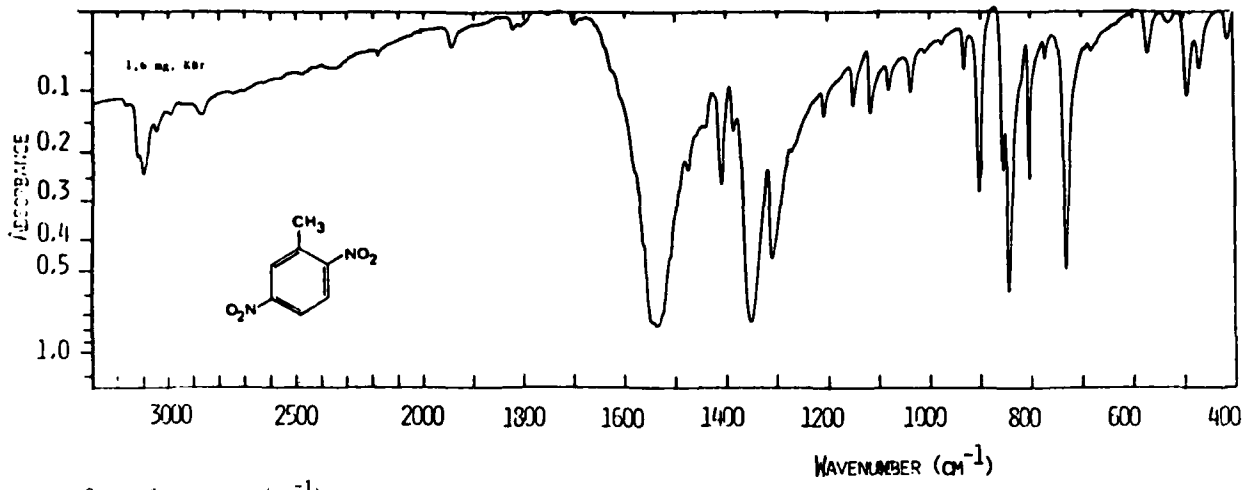


Inlet: GC
Column: OV 225
Ion source: 150 °C



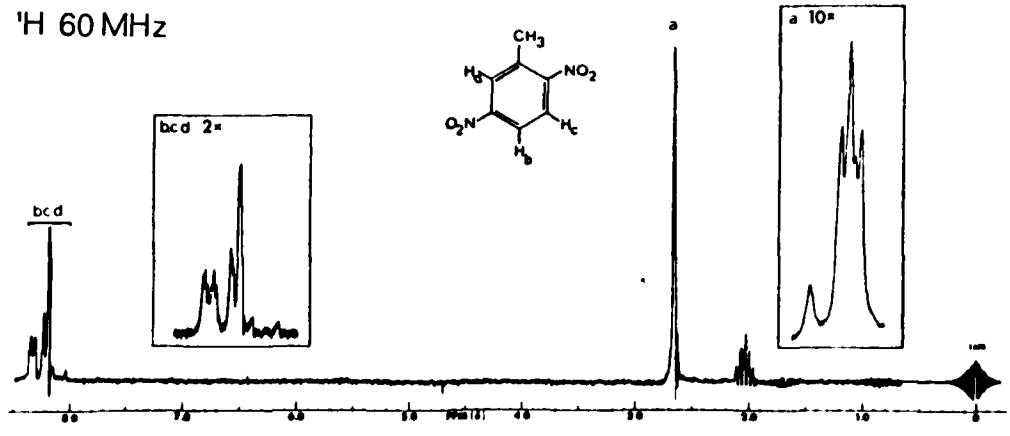
Inlet: GC
Column: OV 225
Ion source: 150 °C

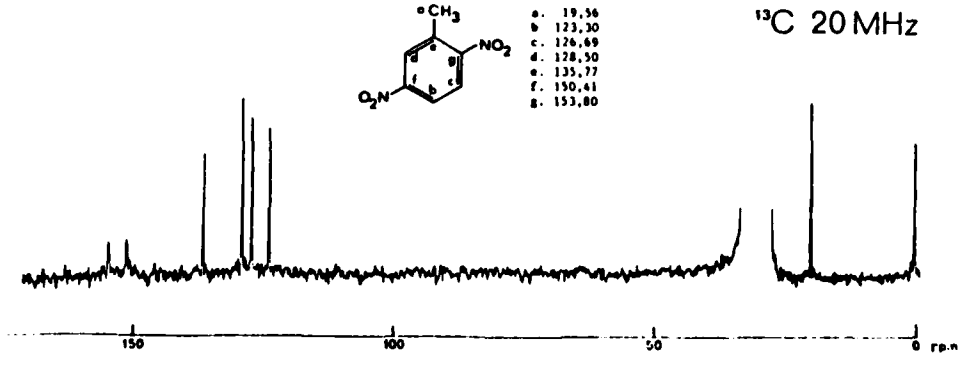
(18)



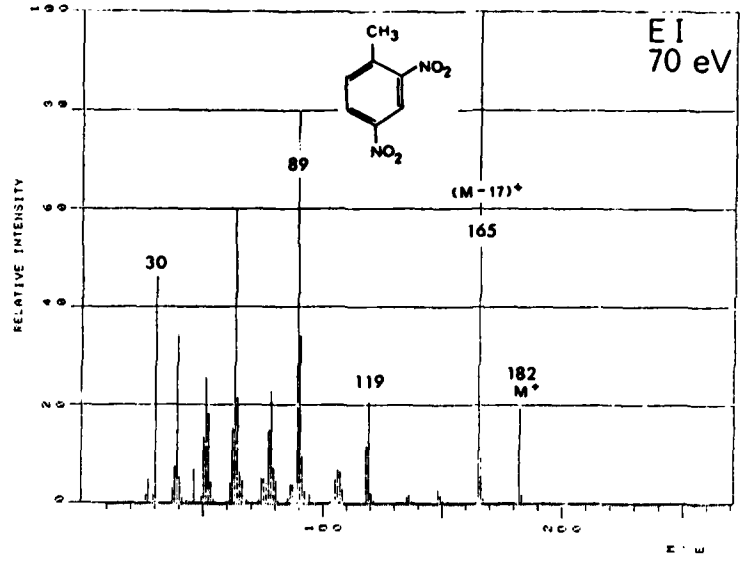
Group frequences (cm^{-1}):

3124, 3100, 3044	C-H arom	1547, 1532, 1523	(C)-NO_2 asym	903 Isol C-H arom
2992	C-H aliph	1350	(C)-NO_2 sym	

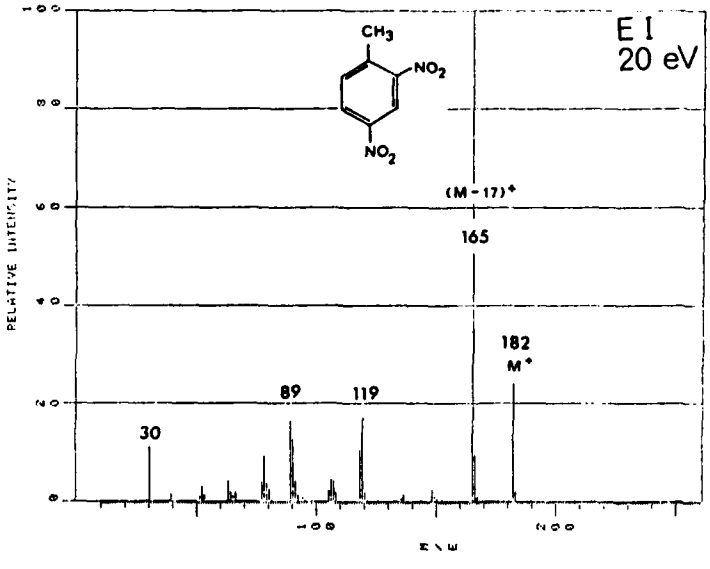




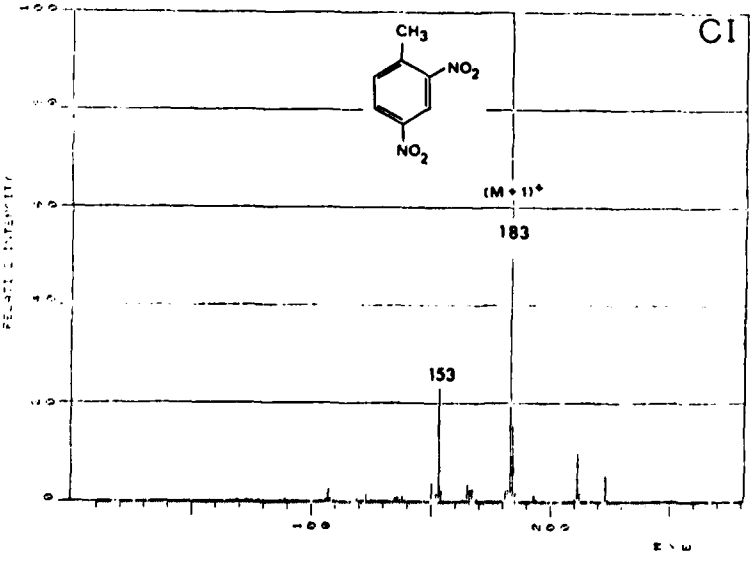
2,4 - DNT



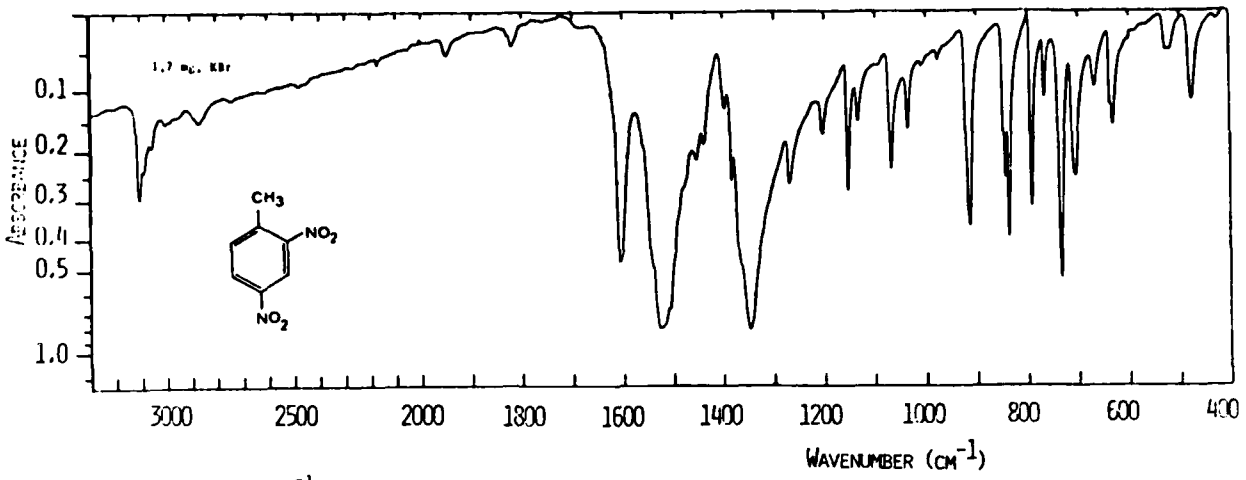
Inlet: GC
Column: OV 225
Ion source: 150 °C



Inlet: GC
Column: OV 225
Ion source: 150 °C

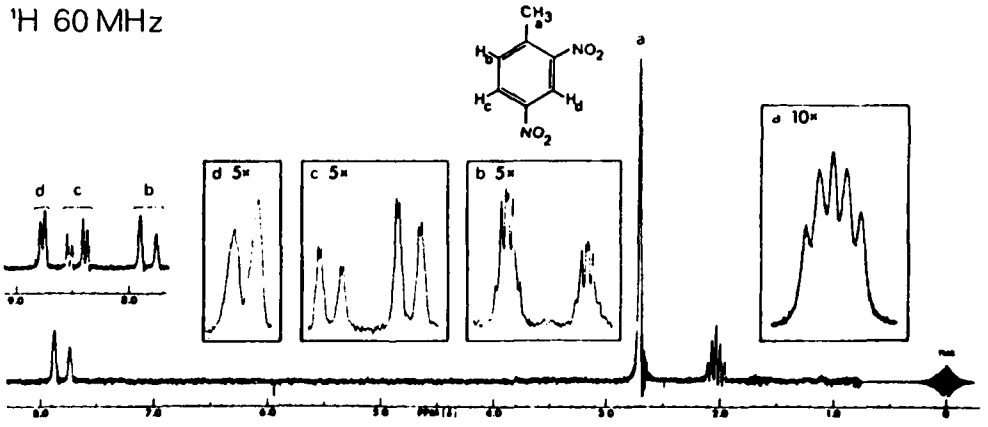


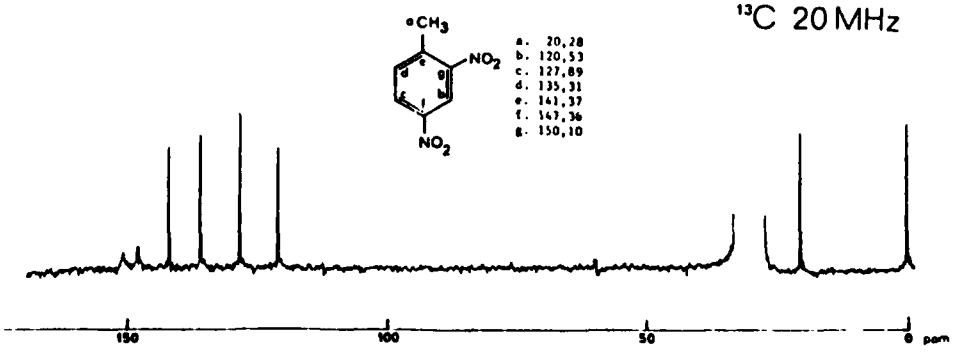
Inlet: GC
Column: OV 225
Ion source: 150 °C



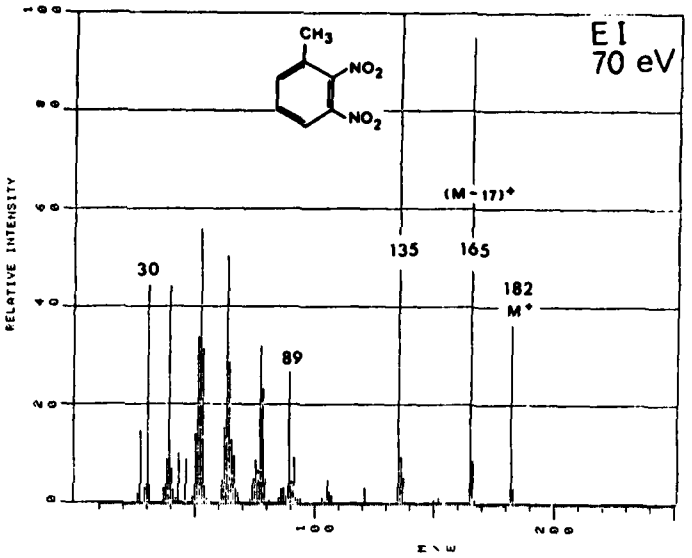
Group frequences (cm^{-1}):

3105, 3085, 3060	C-H arom	1540, 1523, 1509	(C)-NO_2 asym	1070, 913 Isol C-H arom
1608	phenyl	1348	(C)-NO_2 sym	

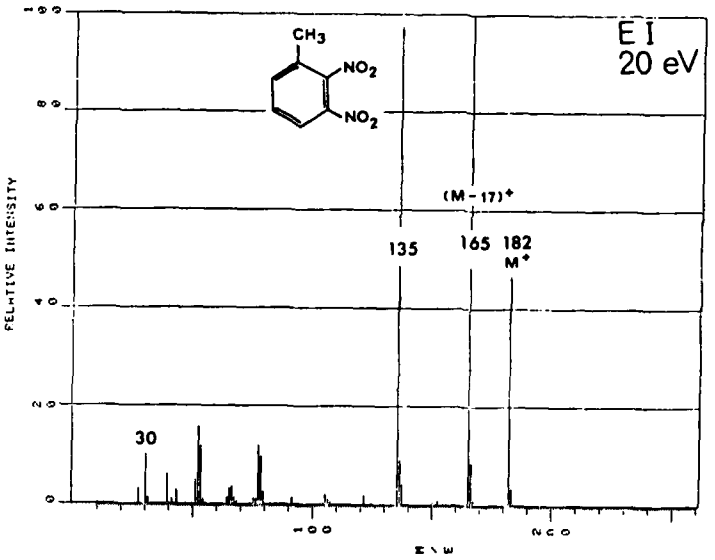




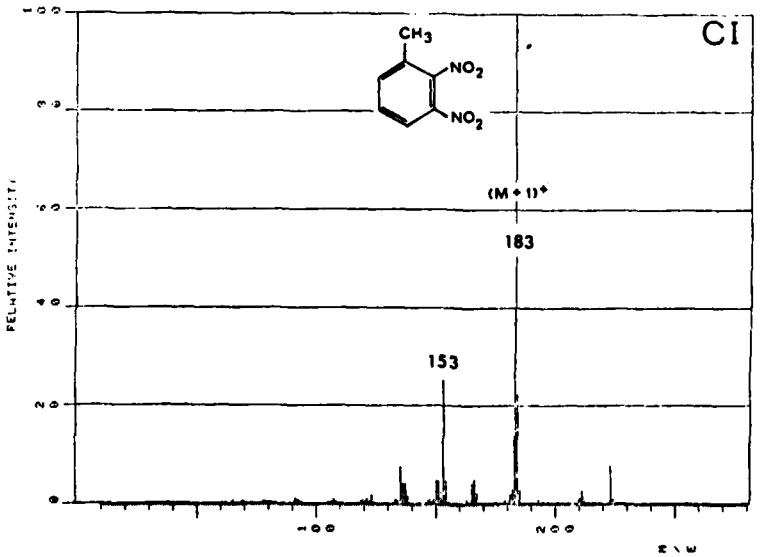
60
2,3 - DNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

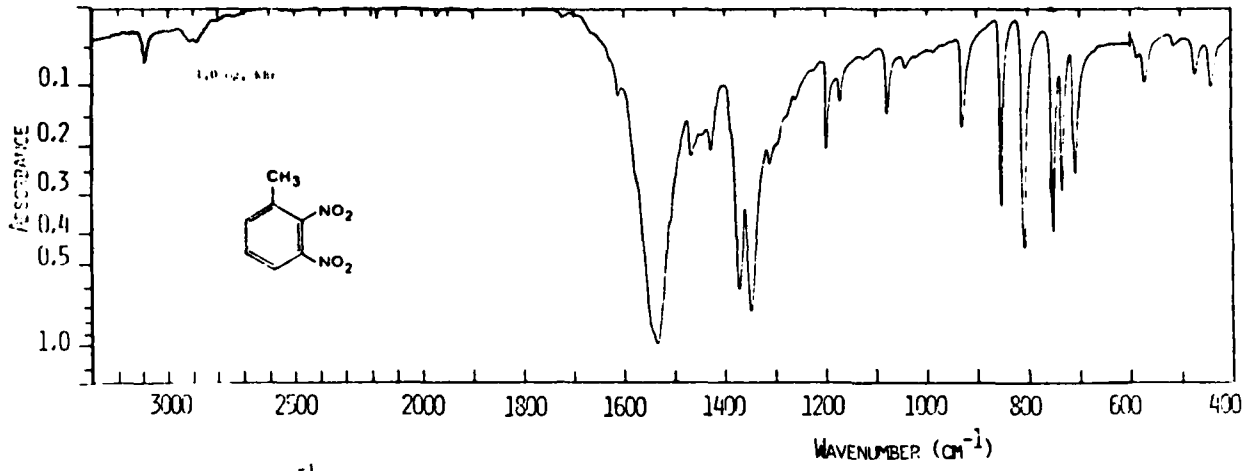


Inlet: GC
Column: OV 225
Ion source: 150 °C



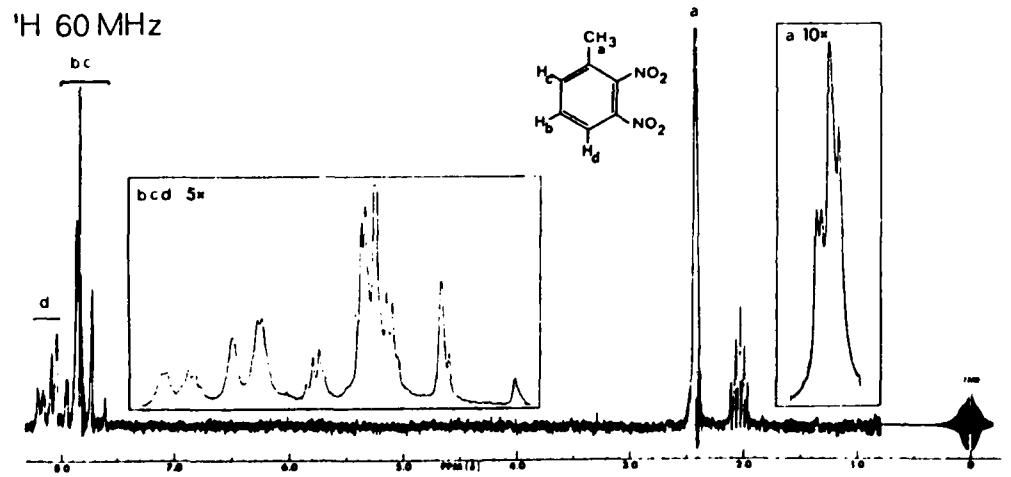
Inlet: GC
Column: OV 225
Ion source: 150 °C

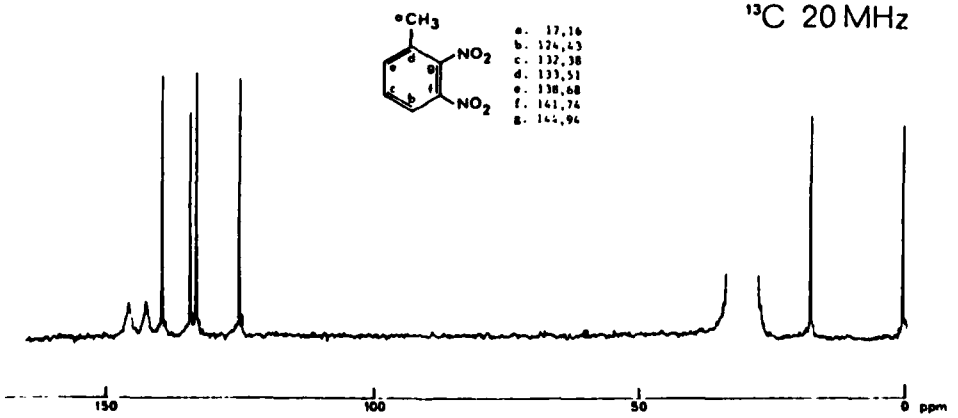
(20)



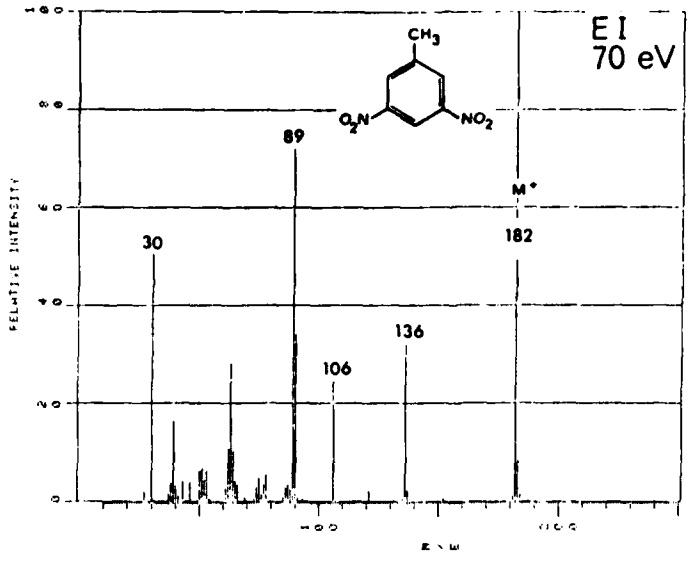
Group frequences (cm^{-1}):

3105	C-H arom	1547, 1535	(C)-NO_2 asym
1610	phenyl	1370, 1350	(C)-NO_2 sym

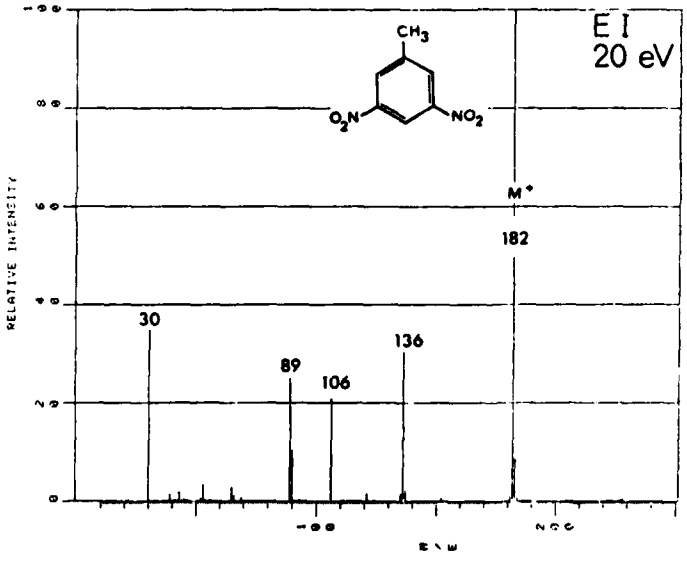




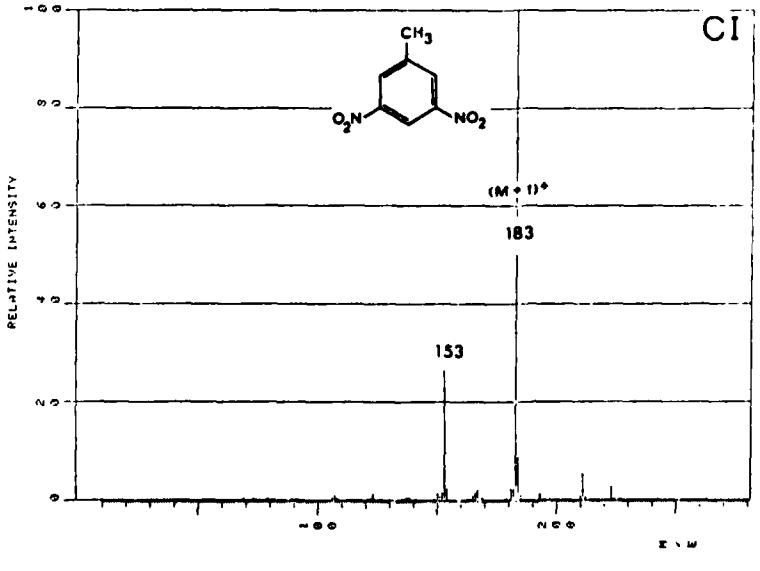
3,5 – DNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

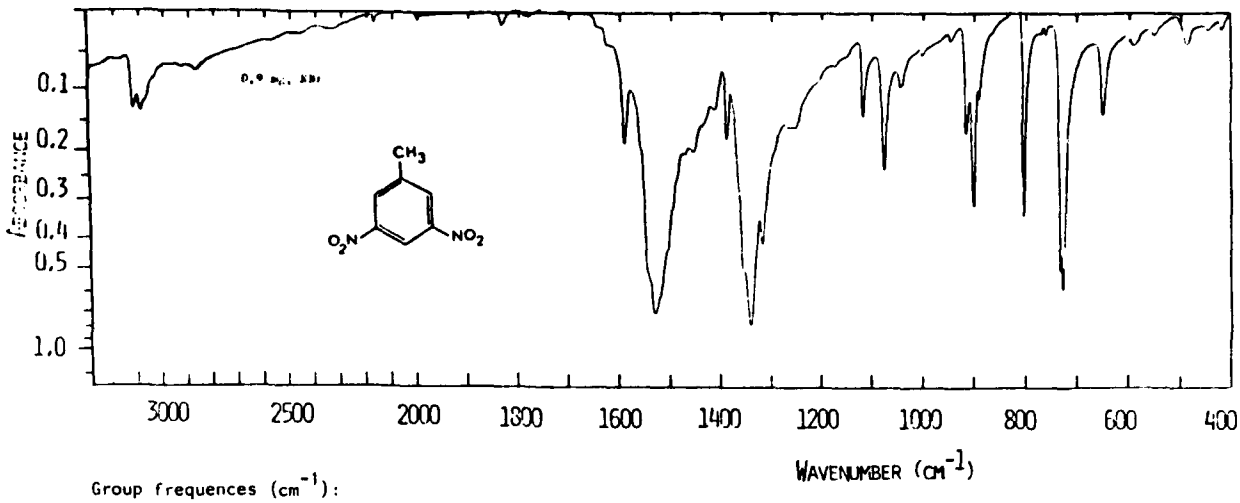


Inlet: GC
Column: OV 225
Ion source: 150 °C



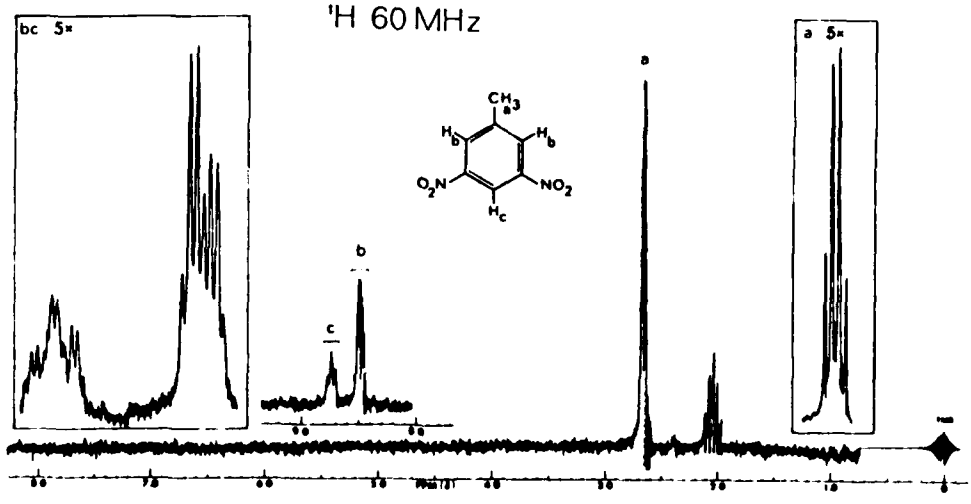
Inlet: GC
Column: OV 225
Ion source: 150 °C

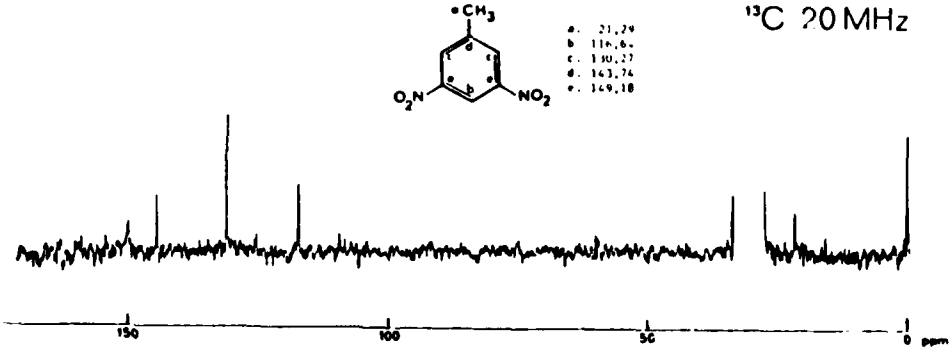
(21)



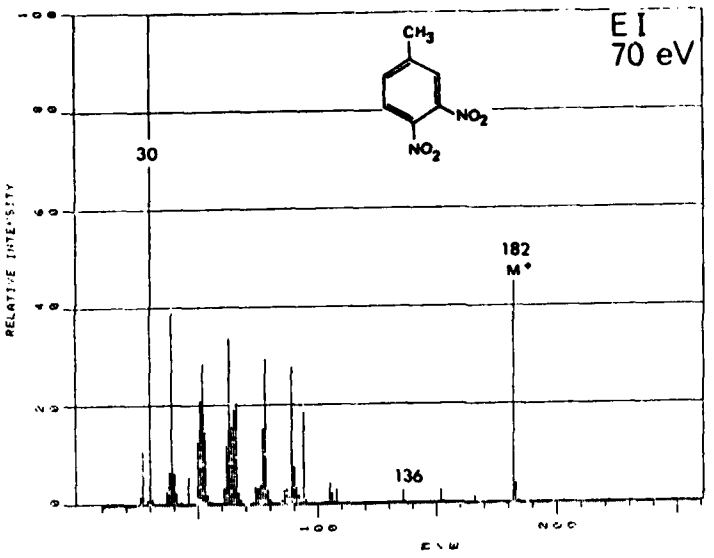
Group frequences (cm^{-1}):

3120, 3088, 3074	C-H arom	1542, 1531, 1523	(C)-NO_2 asym	1078, 918, 902 Isol C-H arom
1590	phenyl	1353, 1341	(C)-NO_2 sym	

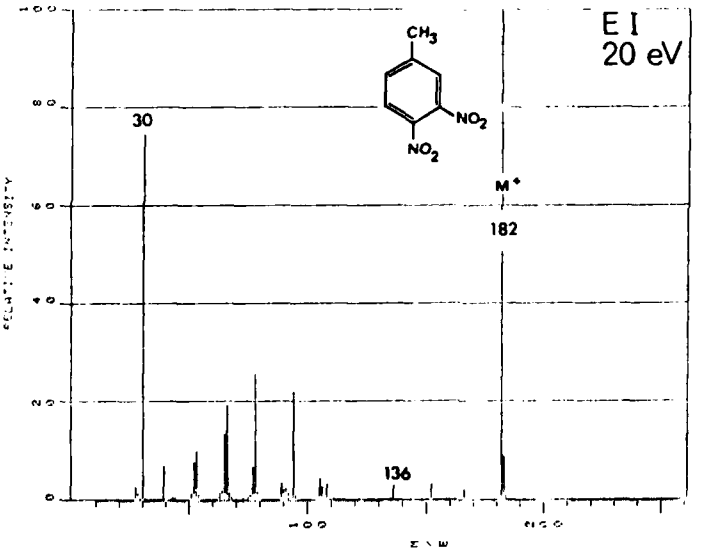




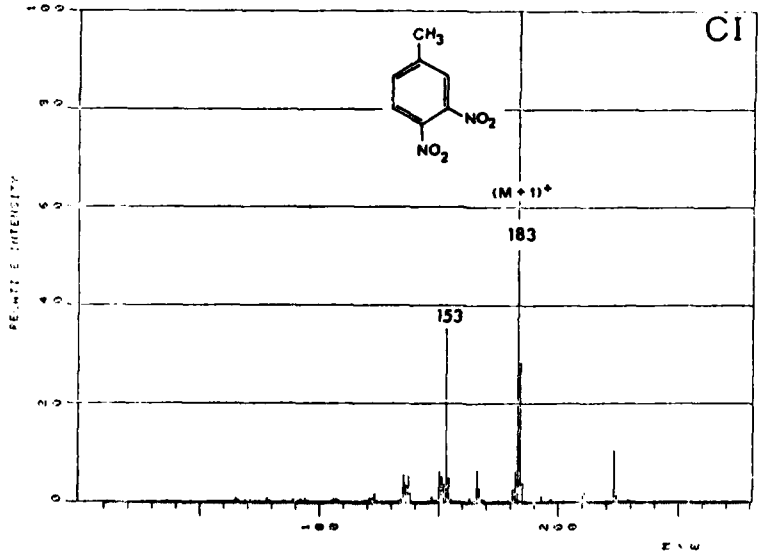
3,4 – DNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

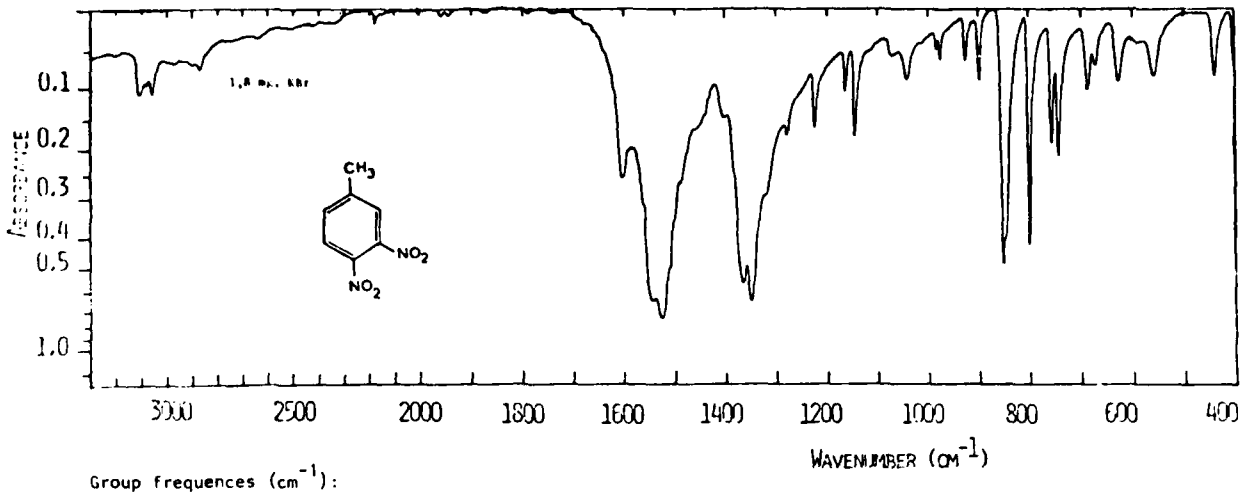


Inlet: GC
Column: OV 225
Ion source: 150 °C



Inlet: GC
Column: OV 225
Ion source: 150 °C

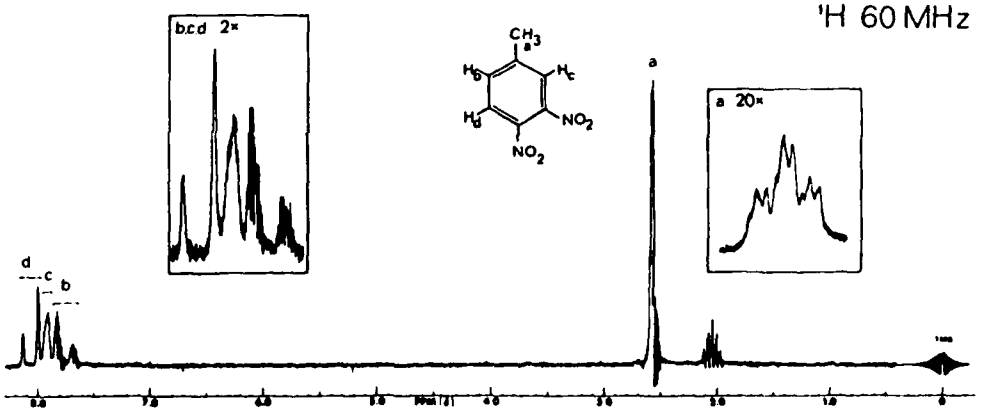
(22)



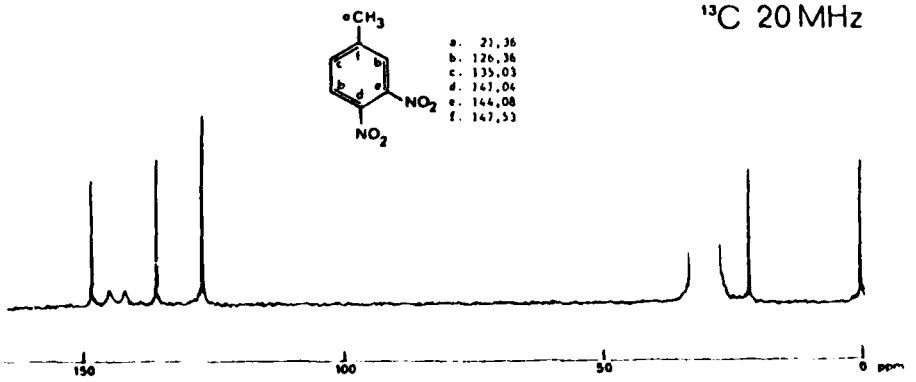
Group frequences (cm^{-1}):

3118, 3078, 3061	C-H arom	1544, 1527	(C)-NO_2 asym	899 Isol C-H arom
1608, 1601	phenyl	1364, 1350	(C)-NO_2 sym	

^1H 60 MHz

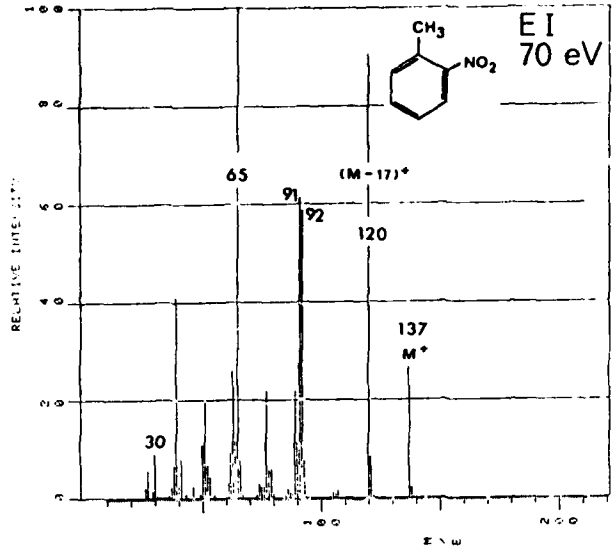


^{13}C 20 MHz

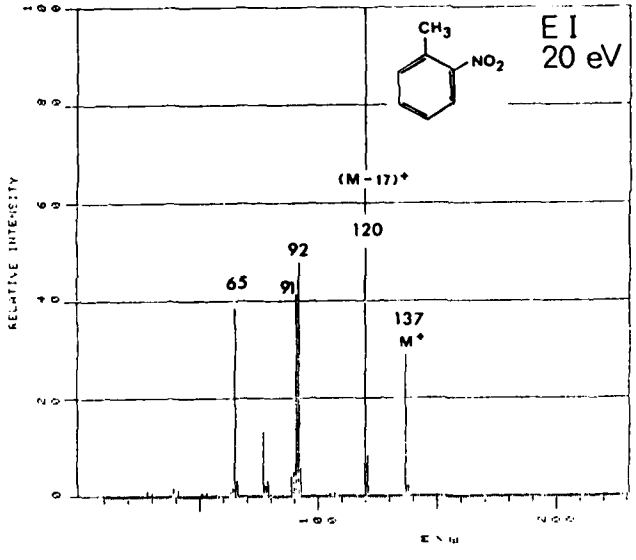


66

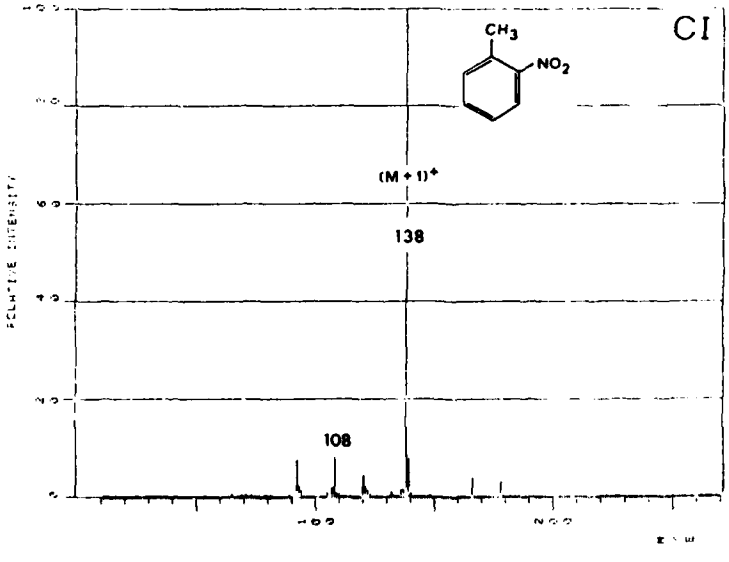
2-MNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

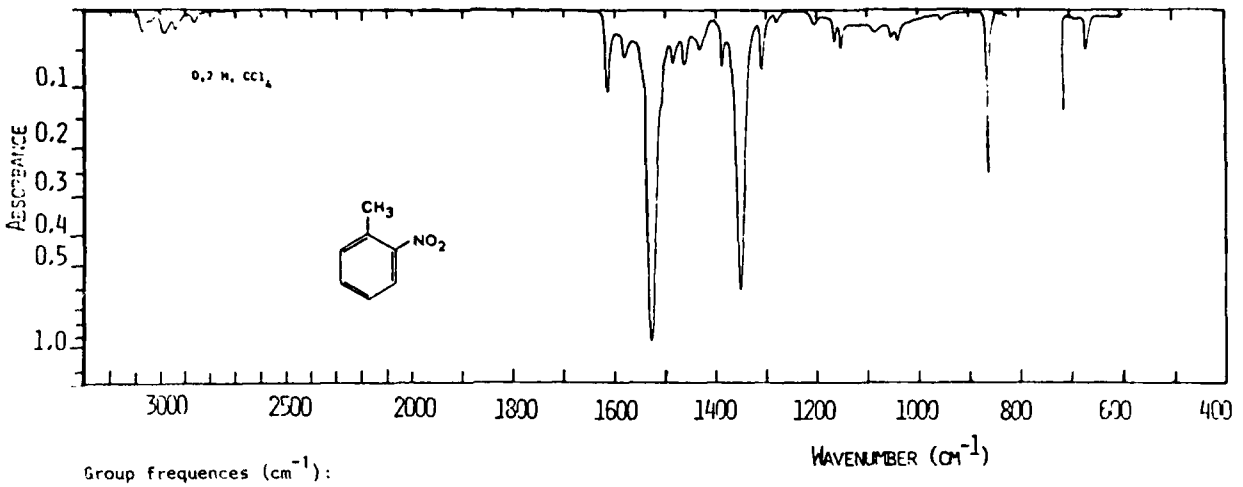


Inlet: GC
Column: OV 225
Ion source: 150 °C



Inlet: GC
Column: OV 225
Ion source: 150 °C

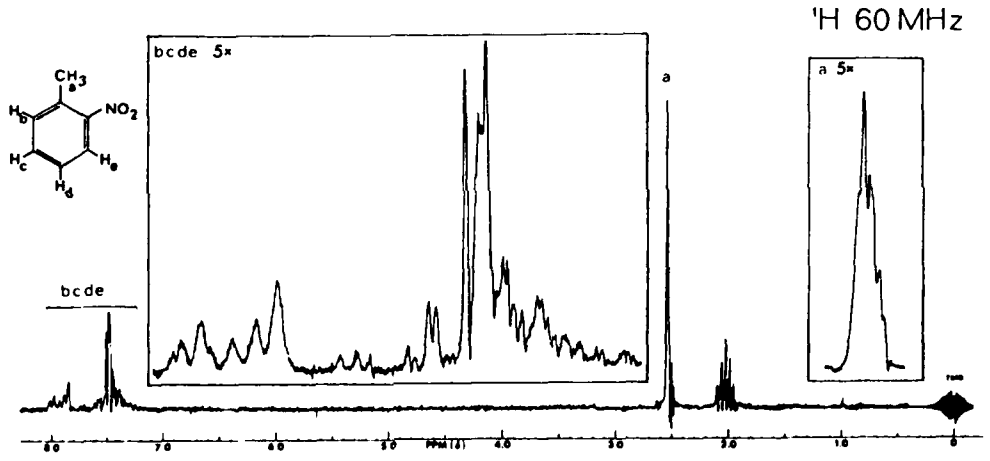
(23)



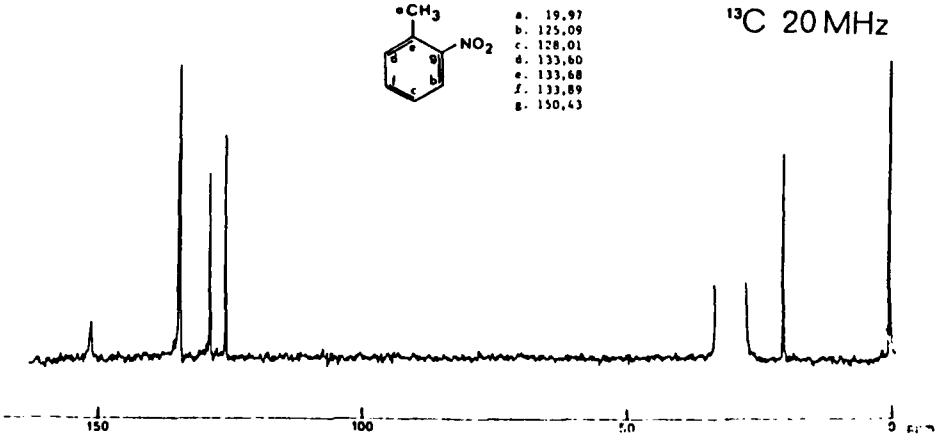
Group frequences (cm^{-1}):

3078	C-H arom	1615	phenyl	1528	(C)-NO_2 asym
2984, 2940	C-H aliph	1580	C-H arom	1351	(C)-NO_2 sym

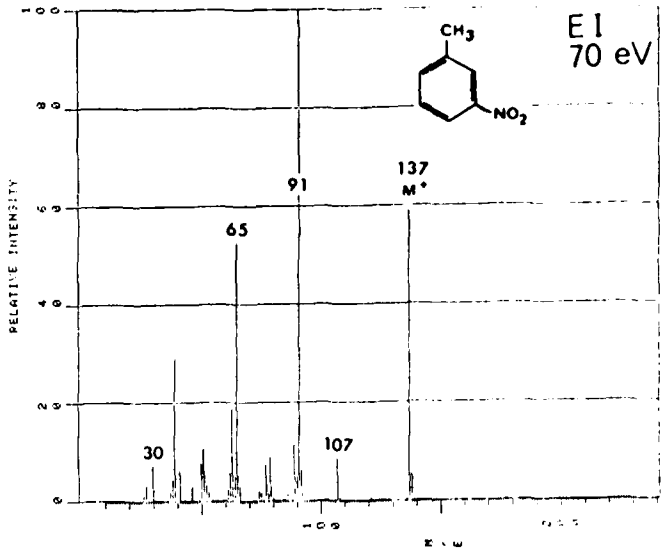
^1H 60 MHz



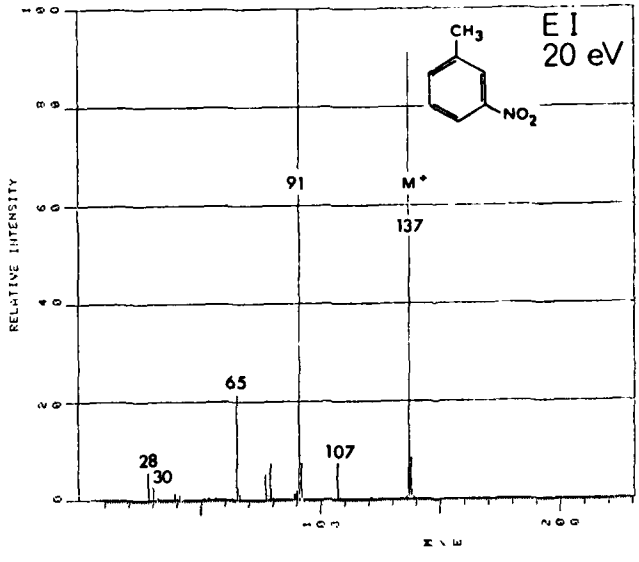
^{13}C 20 MHz



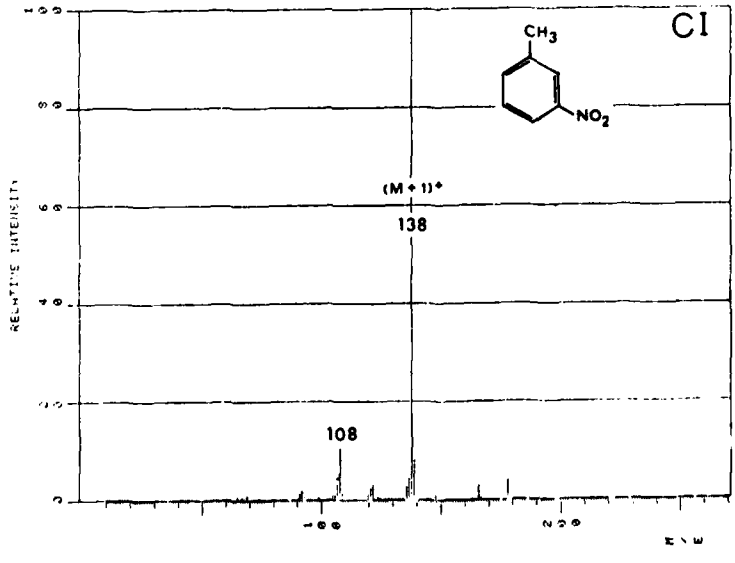
3-MNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

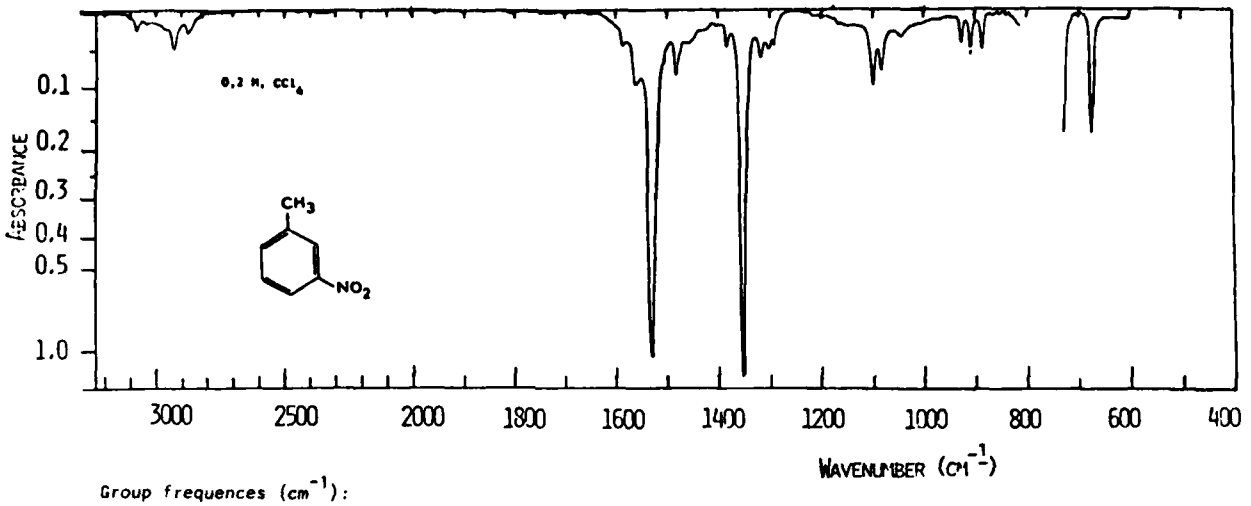


Inlet: GC
Column: OV 225
Ion source: 150 °C



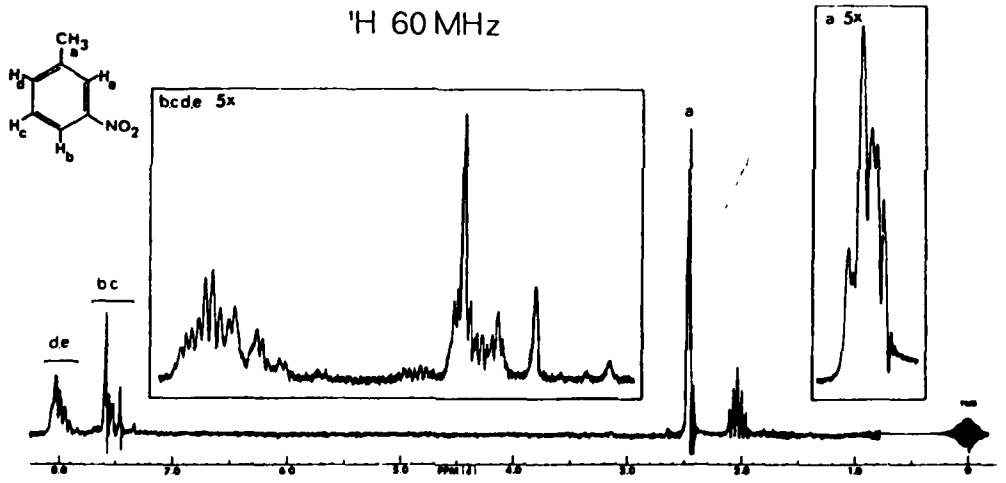
Inlet: GC
Column: OV 225
Ion source: 150 °C

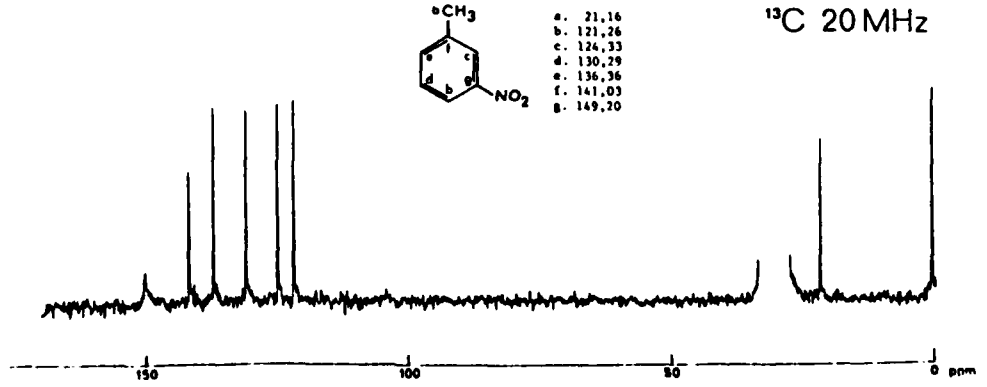
(24)



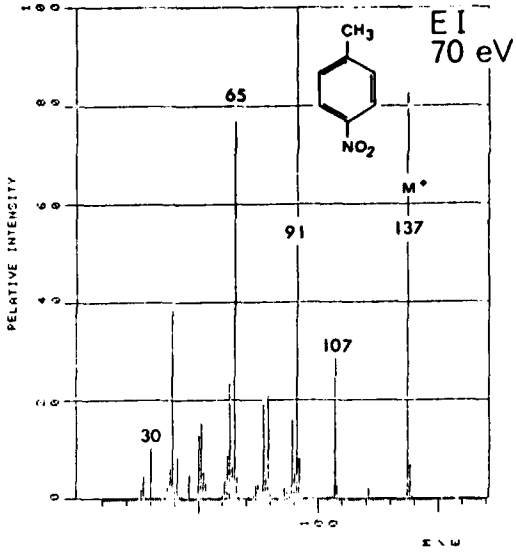
Group frequences (cm^{-1}):

3100, 3078, 3039	C-H arom	1590	phenyl	1354	(C)-NO_2 sym
2932	C-H aliph	1532	(C)-NO_2 asym		

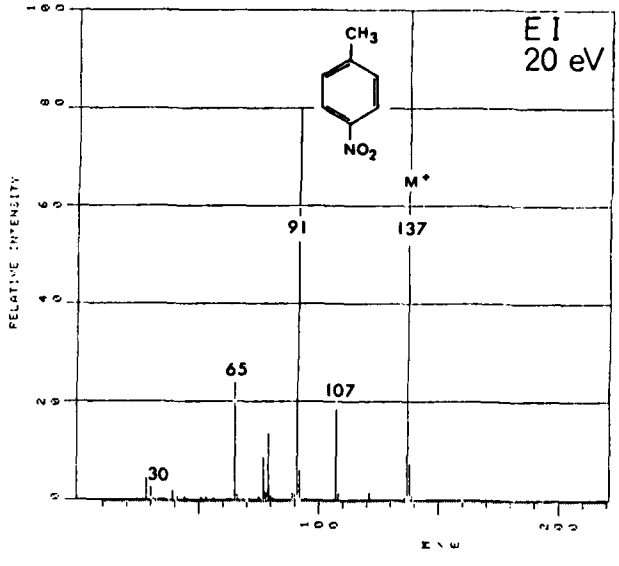




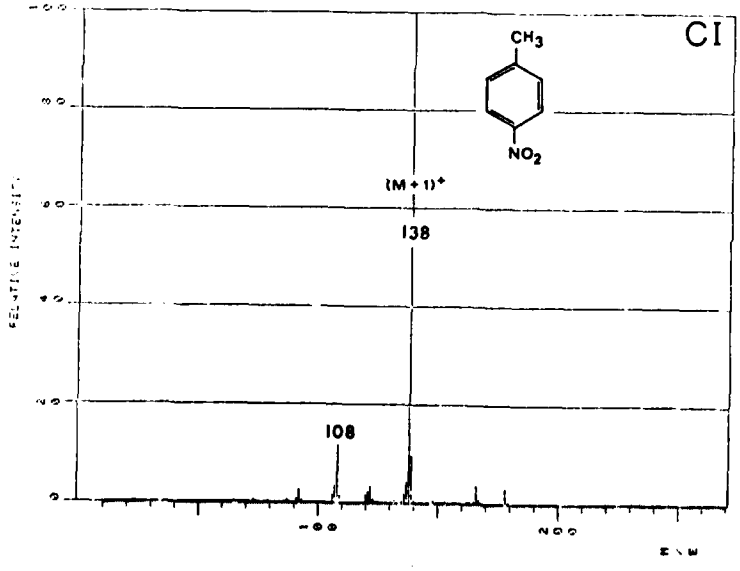
4-MNT



Inlet: GC
Column: OV 225
Ion source: 150 °C

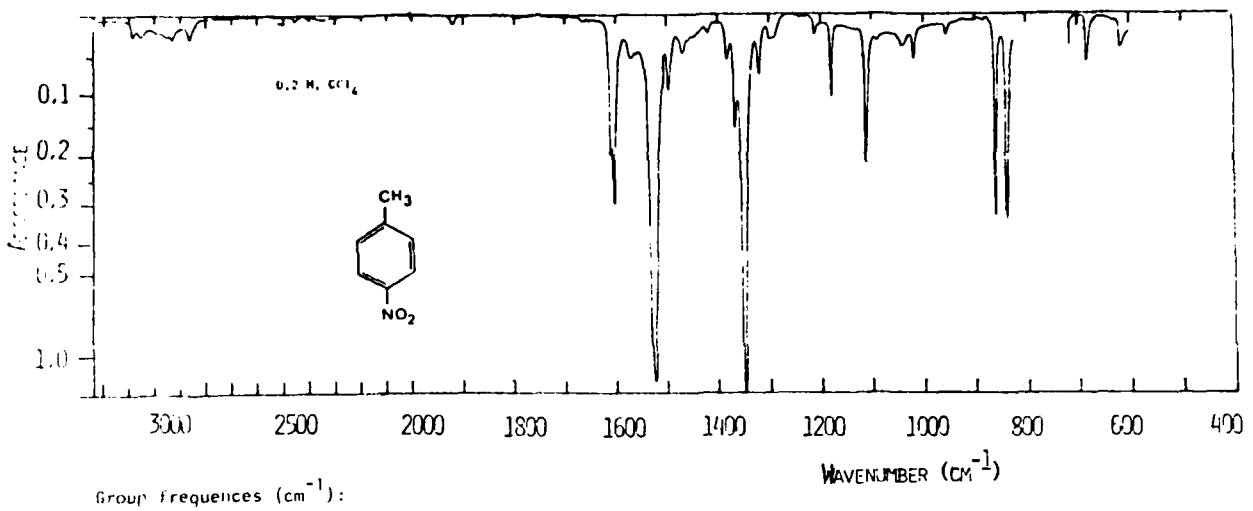


Inlet: GC
Column: OV 225
Ion source: 150 °C



Inlet: GC
Column: OV 225
Ion source: 150 °C

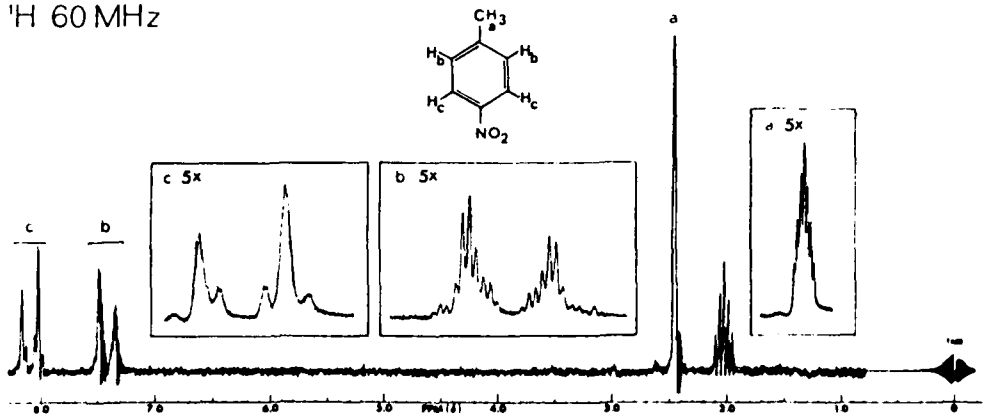
(25)



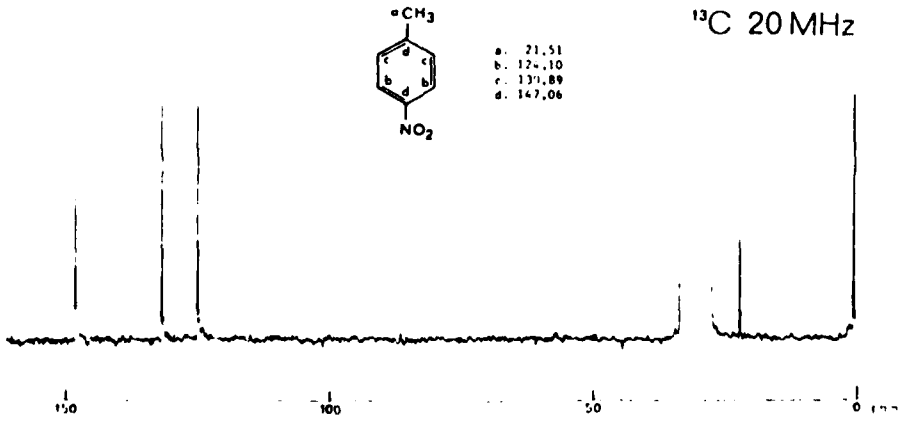
Group Frequences (cm^{-1}):

3084, 3053	C-H arom	1608, 1601	phenyl	1523	(C)-NO_2 asym
2992, 2956, 2936	C-H aliph	1570	C-H arom	1350	(C)-NO_2 sym

^{1}H 60 MHz



^{13}C 20 MHz



COLOUR REACTIONS

Spot tests[16]

The reactions were performed using a sample size of 10 µg dissolved in 50 µl acetone (0.02 %). Two drops of each reactant were added. Ethanol can be chosen instead of acetone if the sample is soluble in this solvent.

Methods of detection

1. Sodium hydroxide

   1 M NaOH

   Colour appears instantly

2. Sodium hydroxide + Griess' reagent

   A. 1 M NaOH
   B. Griess' reagent:
      1 vol 1 % sulphanilic acid in 30 % HAc + 1 vol 0.1 %
      N-(1-naphthyl)-ethylenediaminodihydrochloride in water

   10 min between adding A and B, colour appears in 2 min.

3. Zinc + Griess' reagent

   A. Zinc dust
   B. *Griess' reagent as above*

   Colour appears in 2 min.

4. Zinc + hydrochloric acid + p-dimethylaminobenzaldehyde (DMAB)

   A. Zinc dust
   B. 3 M HCl
   C. Saturated solution of DMAB in benzene.

   Add A + B, boil for 2 min and centrifugate. Transfer the solution to a filter paper and dry. Add C. Colour appears instantly (heating may be necessary).

5. Diphenylamine (DPA) in sulphuric acid

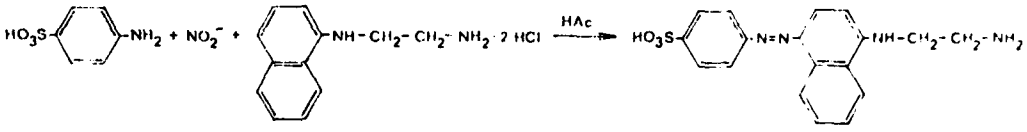
   0.1 % DPA in conc. H_2SO_4

   Colour appears instantly.

Reactions:

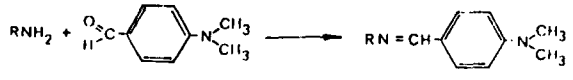
Method 1: Polynitroaromates are transformed to coloured complexes in alkaline media (Meisenheimer complex).

Method 2: Nitrite, liberated by alkali, and Griess' reagent react giving an azo dyestuff as product (within 2 min).



Method 3: Nitrate, liberated by acid, is reduced to nitrite by Zn + acid. The colour reaction is the same as in Method 2.

Method 4: Nitrosubstituents of aromates is reduced to primary amines by Zn + acid. These react with DMAB giving coloured Shiff's bases as products.



Method 5: Nitrate or nitrite, liberated by acid, oxidizes DPA to a blue dyestuff.

Table 6. Colours with spot test reagents.

Detection method / Substance	1	2	3	4	5
1. EGDN	-	Or +	Or ++	-	Bl ++
2. NG	-	Or +++	-"-	-	Bl +++
3. PETN	-	Or + [1]	-"-	-	Bl +++ [1]
4. NC	-	Or +++	Rö + [1]	-	Bl ++ [1]
5. RDX	-	Or +++	Or ++	-	Bl +++ [1]
6. HMX	-	Or ++	Or +	-	- [1]
7. TETR	RöBr +++	RöOr +++ [1]	- [1]	Or +	- [1]
8. AM-PIKR	Gu ++	GuGr ++	-	Gr +	-
9. HNS	RöBr +++	BrGu +	-	Or +	-
10. 1,3,5-TNB	Br +++	Gu ++ [1]	-	Gu +++	-
11. 2,4,6-TNT	RöBr +++	GuBr ++ [1]	-	GuOr ++	-
12. 2,4,5-TNT	RöVi +++	Or ++	-	RöVi ++	-
13. 2,3,6-TNT	BrGu ++	-"-	-	Gu +	-
14. 2,3,5-TNT	Rö ++	-"-	-	Or ++	-
15. 2,3,4-TNT	RöBr +++	-"-	-	Gu +	-
16. 3,4,5-TNT	Vi +++	-"-	-	Gu ++	-
17. 2,6-DNT	Rö + [1]	- [1]	-	Gu +++	-
18. 2,5-DNT	- [1]	- [1]	-	Or +++	-
19. 2,4-DNT	Bl ++ [1] [2]	Rö ++ [1]	-	OrGu +++	-
20. 2,3-DNT	- [1]	- [1]	-	Gu +	-
21. 3,5-DNT	Rö + [1]	Rö ++ [1]	-	GuOr +++	-
22. 3,4-DNT	- [1]	- [1]	-	Gu ++	-
23-25. MNT-er	- [1]	- [1]	-	Gu +++	-
Nitrite	-	Rö +++	Rö +++	-	Bl +++
Nitrate	-	-	Rö +++	-	Bl +++

[1] Valid for 100 µg substance
[2] Colour fades rapidly

Abbreviations:

Bl	blue	Rö	red	-	no colour
Gr	green	Vi	violet	+	faint
Gu	yellow	Br	brown	++	medium
Or	orange			+++	strong

Detection on silica gel layers

The data of reagents and colours given below are cited from a report of Bilson[17]. These reagents have similar underlying reaction mechanisms as the spot tests. The modifications of composition of the reagents, conditioned by the presence of silica gel, will in some cases cause a change in colour shade.

Spotted amounts of sample have been 10 µg. Colours were developed by spray techniques.

Methods of detection

1. No reagents

   a) Daylight
   b)UV-light, 254 nm

2. Alkaline methanol

   10 % KOH in methanol

3. Alkaline methanol + Griess' reagent

   A. Alkaline methanol, see above
   B. Griess' reagent, see spot test reagent 2 B.

   20 min at 105 ^{o}C between A and B.

4. Diphenylamine (DPA)

   1.5 % DPA in methanol

   UV-light, 254 nm, 15 min.

5. Titaniumtrichloride + p-dimethylaminobenzaldehyde (DMAB)

   A. 12.5 % $TiCl_3$ in 15 % HCl
   B. 0.25 % DMAB in 30 % HAc

   a) Air drying between A and B
   b) + heating at 105 ^{o}C during 15 min.

Table 7. Colours on silica gel layers (cit. Bilson[17]).

Detection method / Substance	1a	1b	2	3	4	5a	5b
2. NG	-	-	-	Rö ++	GBr ++	-	-
3. PETN	-	-	-	-"-	-"-	-	-
4. NC	-	Pu (+)	-	-"-	GuGr +	-	-
5. RDX	-	Pu ++	-	-"-	BlG ++	Gu ++	Gu ++
6. HMX	-	-"-	-	-"-	Gu +	-	-
7. TETR	Gu ++	BrPu ++	RöBr ++	GuBr ++	Br ++	GuOr ++	Br ++
8. AM-PIKR	-"-	-"-	Gu ++	Gu ++	Gu ++	Gu ++	OrBr ++
10. TNB	-	Pu ++	RöBr ++	RöBr +	PuBr ++	GuOr ++	GuBr ++
11. TNT	-	-"-	-"-	RöBr ++	Br +	Gu ++	-"-
19. 2,4-DNT	-	-"-	-	RöBr +	Br +	-"-	Gu ++
23. 2-MNT	-	-"-	-	-	-	PuBr ++	PuBr ++

Abbreviations:

Bl	blue	Gu	yellow	-	no colour
Br	brown	Or	orange	(+)	very pale
G	grey	Pu	purple	+	pale
Gr	green	Rö	red	++	distinct

THIN LAYER CHROMATOGRAPHY

Thin layer chromatography is used in quantitative and qualitative analysis[2].

The substances were chromatographed on 0.25 mm silica gel layer containing fluoroscence indicator (Silica Gel F-254, Merck No. 5715). Spotted amount was 2 µl of a 0.1 % solution (2 µg) with the exception of the nitric esters where sample amount was 10 - 20 times bigger.

The eluents were chosen according to Bilson[17]. The experimental conditions regarding activity of layers and saturation of chamber, however, were different. Found and cited R_f-values in table 8 therefore correspond to those maximum and minimum values one can expect for silica layers. The travel distance of solvents were 10 cm.

One example of a two dimensional chromatogram is given in figure 1.

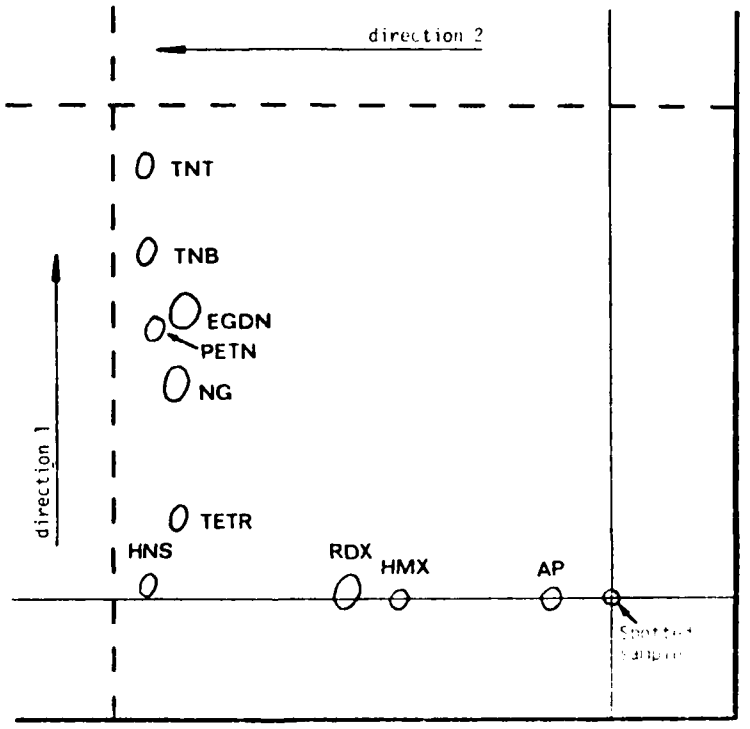


Figure 1. Twodimensional chromatogram of explosives (no 1 - 3, 5 - 11). Layer: Silica Gel F-254. Travel distance: 10 cm.

Direction 1: Light petroleum (60 - 80 ^{o}C)/Diisopropylether 25:75

Direction 2: Benzene/Acetone 70:30

Table 3. R_f-values on silica gel layers.

A. Layers not activated. Chamber not lined with filterpaper.

Substances \ Eluents		1	2	3	4	5	6	7	8	9	10	Two dimen 1	2
3	AM-PIKR	0	0	0	0	0,11	0,14	0,38	0,36	0,74	0,97	0	0,12
6	HMX	0	0	0,01	0,02	0,36	0,25	0,56t	0,66	0,85	0,76u	0	0,43
5	RDX	0	0,01	0,03	0,10	0,48	0,72	0,64	0,71	0,80	0,81	0,01	0,53
9	HNS	0	0	0	0,22	0,95	0,80	0,96	0,97	0,99	0,95	0,02	0,93
7	TETR	0	0,03	0,18	0,43	0,88	0,82	0,89	0,94	0,98	0,92	0,17	0,87
2	NG	0,01	0,12	0,43	0,66	0,87	0,91	0,88	0,93	0,98	0,92	0,42	0,87
3	PETN	0	0,13	0,66	0,66	0,93	0,91	0,93	0,94	0,99	0,94	0,55	0,92
1	EGDN	0,03	0,23	0,42	0,73	0,87	0,92	0,90	0,92	0,97	0,93	0,58	0,85
10	TNB	0	0,26	0,58	0,63	0,94	0,87	0,94	0,96	0,99	0,93	0,70	0,92
11	TNT	0	0,45	0,71	0,73	0,96	0,90	0,95	0,96	0,99	0,94	0,87	0,92

Eluents in twodimensional chromatogram: Direction 1. Light petroleum (60 - 80 °C)/Diisopropylether 25:75

Direction 2. Benzene/Acetone 70:30

ntin. Table 8.

B. Layers activated 1 hour at 105 °C, chamber lined with filter paper (cit Bilson[17]).

Substance \ Eluent	1	2	3	4	5	6	7	8	9	10
8 AM-PIKR	0	0	0	0	0,13	0,17	0,25	0,29	0,53	0,80
6 HMX	0	0	0	0	0,31	u	u	0,46	0,59	u
5 RDX	0	0	0,02	0,07	0,42	0,52	0,48	0,53	0,57	0,64
9 HNS	-	-	-	-	-	-	-	-	-	-
7 TETR	0	0,03	0,13	0,33	0,71	0,70	0,72	0,75	0,77	0,77
2 NG	0	0,06	0,31	0,47	0,70	0,82	0,70	0,71	0,76	0,78
3 PETN	0	0,08	0,51	0,49	0,75	0,84	0,76	0,79	0,76	0,77
1 EGDN	-	-	-	-	-	-	-	-	-	-
10 TNB	0	0,17	0,42	0,47	0,76	0,81	0,77	0,81	0,78	0,76
11 TNT	0	0,28	0,52	0,54	0,76	0,83	0,79	0,79	0,78	0,76

t = tailing u = long

Eluents (mixtures in v/v)

1. Light petroleum, 60-80 °C
2. Light petroleum, 60-80 °C/Diisopropylether 70:30
3. Light petroleum, 60-80 °C/n-Butyl acetate 80:20
4. Benzene
5. Benzene/Acetone 75:25
6. Dichloromethane/Acetic acid 90:10
7. Benzene/Methyl alcohol/Acetic acid 90:16:8
8. Benzene/Acetone/Methyl alcohol 70:20:10
9. Dioxan/benzene/Acetic acid 75:27:12
10. Methyl alcohol

Purity:

Acetone pa	Merck no 14
Benzene pa	Merck no 1783
n-Butyl acetate, 98-100 % purum	Kebo no 25477
Diisopropylether, 66-69 °C, ca 97 %	Kebo no 23723
Dichloromethane, 98-100 %	Kebo no 13090
Dioxan pa	Merck no 9671
Methyl alcohol pa	Merck no 6009
Light petroleum, 60-80 °C, pa	Merck no 1774
Acetic acid, 99-100 %, pa	Riedel-de Haen no 32209

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